universite PARIS-SACLAY

# Biomechanical characterization of the tongue-food interface during the course of oral processing

*Caractérisation biomécanique de l'interface langue-aliment au cours du processus oral*

**Thèse de doctorat de l'université Paris-Saclay**

Ecole doctorale N°581, Agriculture, alimentation, biologie, environnement et santé (ABIES)

Spécialité de doctorat: Génie des Aliments

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Référent : AgroParisTech

**Thèse présentée et soutenue à Paris-Saclay, le 09/07/2021, par**

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**Thèse de doctorat**

NNT : 2021UPASB027

Doctoral Thesis

# Biomechanical characterization of the tongue-food interface during the course of oral processing

*Every sentence I utter must be understood not as an affirmation, but as a question.*

*Niels Bohr*

# An overview

*The quest to develop technologies to better understand and model consumer's sensorial perceptions of food is of paramount interest to meet the challenges of food transitions. Food oral processing and associated biomechanics play an important role in the diversity of the dynamic perceptions of food texture. This doctoral project was aimed to understand the biomechanics of the tongue-food interface by designing novel in-vitro oral processing set-ups that take into account targeted oral physiological variables. Furthermore, original non-destructive and non-invasive methods such as ultrasound-based techniques were developed to provide real-time analysis of the tongue-food-palate system. This thesis hence involved a multidisciplinary approach which employed expertise in biomechanics, acoustics, food science and sensory analysis.*

*At first, ultrasound equipped biomimetic set-up was developed which helped us to investigate the deformation of gelatin and/or agar based gels over artificial tongue models during uniaxial compression. The model tongues were fabricated from polyvinyl alcohol (PVA), which helped to mimic the real physiological characteristics of human tongue like rigidity and surface roughness. Two ultrasound parameters (i) reflection coefficient and (ii) time of flight) were identified as suitable for analyzing tongue-food interface coupling and bulk deformation respectively. The results showed that both tongue properties (rigidity, roughness) and food properties (rigidity and syneresis) were critical. The ultrasound method was then further developed to address not only the tongue and its interface with food, but also the heterogeneities present in food. This work involved developing more complex food models (bilayers of agar/gelatin gels) which were studied for decoding the perception of textural contrast in heterogeneous foods.*

*The second part of this doctoral project was aimed to develop a new oral processing set-up that could perform more complex and realistic sequences of motions (combining compression and shearing) between a PVA tongue mimic and an aluminum plate mimicking the hard palate. This set-up was equipped with three-axis force sensors in order to evaluate friction forces. Cottage cheese with and without suspended particles (varied size, shape and rigidity) was used as model food in the experiments, making it possible to introduce heterogeneities approaching the perception thresholds. The feasibility of the set-up was tested by measuring how friction coefficient values were affected by food properties (viscosity, particle presence), tongue properties (surface roughness, bulk rigidity), and operational parameters (normal stress,*

*shearing velocity). The measurements with the set-up were found to be repeatable, confirming its reliability. Increases in the surface roughness and bulk rigidity of the tongue mimic led to a pronounced augmentation of the friction coefficient values. The friction coefficient values also showed dependence on normal stress, increase in normal stress led to significant decrease in friction. Moreover, friction notably increased between the lowest and highest shearing velocities.*

*Sensory analysis work was also carried out in order to be able to relate the mechanical phenomena observed during the experimental studies carried out in the two parts of the thesis with the texture perceptions quantified by trained panels. The results of the thesis confirm that beyond the physico-chemical characteristics of food, it is essential to take into account the characteristics of the tongues (as well as their variability) in order to fully understand the mechanical interactions which likely affect the sensory perception of texture by consumers.*

*This PhD project was hence carried out with the aim to contribute towards biomechanical characterization of the tongue-food interface through different novel in vitro approaches and has presented exciting observations which will make us advance in understanding the phenomena behind texture perception. The work also opens up exiting challenges for food oral processing community in terms of bridging further the gap between real oral physiology and instrumental methods especially on key aspects like tongue roughness, stiffness and wettability. These different perspectives will thus allow to lift the technical and scientific barriers to study the mechanisms responsible for the perception of texture directly on the consumer.*

# Résumé

*La recherche de technologies permettant de mieux comprendre et de modéliser les perceptions sensorielles des aliments par les consommateurs est d'un intérêt primordial pour relever les défis des transitions alimentaires. Le processus oral des aliments et la biomécanique associée jouent un rôle important dans la diversité des perceptions dynamiques de la texture des aliments. Ce projet de doctorat avait pour but de comprendre la biomécanique de l'interface langue-aliment en concevant de nouveaux modèles in-vitro du traitement oral qui prennent en compte des variables physiologiques orales spécifiques. En outre, des méthodes originales non destructives et non invasives, telles que des techniques fondées sur les ultrasons, ont été développées pour fournir une analyse en temps réel du système langue-aliment-palais. Cette thèse a donc impliqué une approche multidisciplinaire en faisant appel à des compétences en biomécanique, en acoustique, en sciences alimentaires et en analyse sensorielle.*

*Dans un premier temps, un dispositif biomimétique équipé d'ultrasons a été mis au point et a permis d'étudier la déformation de gels de gélatine et/ou d'agar sur des modèles de langues artificielles lors d'une compression uniaxiale. Les modèles de langues ont été fabriqués à partir d'alcool polyvinylique (PVA), ce qui a permis d'imiter les caractéristiques physiologiques réelles de la langue humaine, comme la rigidité et la rugosité de surface. Deux paramètres ultrasonores ((i) le coefficient de réflexion et (ii) le temps de vol) ont démontré être appropriés pour analyser respectivement le couplage entre la langue et l'aliment, et la déformation du volume. Les résultats ont montré que les propriétés de la langue (rigidité, rugosité) et celles de l'aliment (rigidité et synérèse) étaient importantes. La méthode ultrasonore a ensuite été développée pour aussi prendre en compte les hétérogénéités présentes dans les aliments (en plus des hétérogénéités de l'interface langue-aliment). Ce travail a impliqué le développement de modèles alimentaires plus complexes (gels bicouches d'agar/gélatine) qui a permis de décoder la perception du contraste de texture au sein d'aliments hétérogènes.*

*La deuxième partie de ce projet doctoral visait à développer un nouveau dispositif biomimétique du processus oral capable d'effectuer des séquences de mouvements plus complexes et réalistes (combinant compressions et cisaillements) entre la langue artificielle en PVA et une plaque d'aluminium imitant le palais dur. Ce montage a été équipé de capteurs de force à trois axes afin d'évaluer les forces de friction. Du fromage blanc avec et sans particules en suspension (dont la taille, la forme et la rigidité varient) a été utilisé comme aliment modèle dans les expériences, ce qui a permis d'introduire des niveaux d'hétérogénéités proches du seuil*

*de perception. La faisabilité du montage a été testée en mesurant comment les valeurs des coefficients de friction étaient affectées par les propriétés de l'aliment (viscosité, présence de particules), les propriétés de la langue (rugosité de surface, rigidité) et les paramètres opérationnels (contrainte normale, vitesse de cisaillement). Les mesures effectuées avec le dispositif se sont révélées reproductibles, confirmant sa fiabilité. L'augmentation de la rugosité et de la rigidité de la langue ont conduit à une augmentation prononcée des valeurs du coefficient de friction. Ces valeurs de coefficient de friction ont également montré être dépendantes de la contrainte normale dont l'augmentation a conduit à une diminution significative de la friction. Il est aussi intéressant de noter que le frottement a augmenté de façon notable entre les vitesses de cisaillement les plus faibles et les plus élevées.*

*Des travaux d'analyse sensorielle ont également été réalisés pour mettre en relation les perceptions de texture quantifiées par des panels entraînés avec les phénomènes mécaniques observés lors des études expérimentales menées dans les deux parties de la thèse. Les résultats de la thèse confirment qu'au-delà des caractéristiques physico-chimiques des aliments, il est essentiel de prendre en compte les caractéristiques de la langue (ainsi que leur variabilité) afin de bien comprendre les interactions mécaniques qui sont susceptibles d'affecter la perception sensorielle de la texture par les consommateurs.*

*Ce projet de doctorat a donc été effectué dans le but de contribuer à la caractérisation biomécanique de l'interface langue-aliment par le biais de différentes approches in vitro novatrices et a présenté des observations qui nous permettront de progresser dans la compréhension des phénomènes à l'origine de la perception de la texture. Ces travaux ouvrent également la voie à de nouveaux défis pour la communauté de la transformation orale des aliments en termes de réduction de l'écart entre la physiologie orale réelle et les méthodes instrumentales, en particulier sur des aspects clés tels que la rugosité, la rigidité et la mouillabilité de la langue. Ces différentes perspectives permettront ainsi de lever les barrières techniques et scientifiques pour étudier les mécanismes responsables de la perception de la texture directement sur la bouche du consommateur.*

# Scientific Communications

## Peer reviewed articles

1. A new biomimetic set-up to understand the role of the kinematic, mechanical, and surface characteristics of the tongue in food oral tribological studies.

   **Authors:** Rohit Srivastava, Véronique Bosc, Frédéric Restagno, Carole Tournier, Paul Menut, Isabelle Souchon, Vincent Mathieu.

   Hydrocolloids (Volume 115, June 2021, 106602)
   https://doi.org/10.1016/j.foodhyd.2021.106602

2. Ultrasound monitoring of a deformable tongue-food gel system during uniaxial compression–an in vitro study.

   **Authors:** Rohit Srivastava, Mathieu Mantelet, Anne Saint-Eve, Jean-Luc Gennisson, Frédéric Restagno, Isabelle Souchon, Vincent Mathieu.

   Innovative Food Science and Emerging Technologies (Volume 70, June 2021, 102695)
   https://doi.org/10.1016/j.ifset.2021.102695

3. Texture contrast: Ultrasonic assessment of bi-layer gel deformation during compression on a bio-mimicking tongue.

   **Authors:** Rohit Srivastava, Markus Stieger, Elke Scholten, Isabelle Souchon, Vincent Mathieu.

   Current Research in Food Science (Volume 4, 2021, 449-459)
   https://doi.org/10.1016/j.crfs.2021.06.004

4. Real time ultrasound assessment of contact progress between food gels and tongue mimicking surfaces during a compression.

   **Authors:** Mathieu Mantelet, Rohit Srivastava, Frédéric Restagno, Isabelle Souchon, Vincent Mathieu.

   Food Hydrocolloids (Volume 109, December 2020, 106099)
   https://doi.org/10.1016/j.foodhyd.2020.106099

## Presentations

1. Application of quantitative-ultrasounds on a biomimetic oral processing setup to analyze the evolution of food-tongue interface in relation with texture perception. 32nd European Federation of Food Science and Technology Conference. November 6th - 8th, 2018 - Nantes, France. [Oral]

2. Application of ultrasound for comprehending the texture perceptions resulting from the mechanical breakdown of food on the tongue surface. 13th Pangborn Sensory Sensory Science Symposium. July 28th - August 1st, 2019 - Edinburgh, Scotland. [Oral]

3. Ultrasound based mechanical characterization of the tongue-food system during the course of uniaxial compressions. Webinar on Rheology and Food structuring & destructuring- Groupe français de Rhéologie – March 18th, 2021- Online. [Oral]

4. Potential of a newly developed biomimetic setup to better integrate the kinematic, mechanical and surface characteristics of tongue in oral tribological studies of foods. 5th International Conference on BioTribology. April 26th – 28th, 2021 – Online. [Oral]

5. Texture contrast: Ultrasonic quantification of bulk deformations during compression of bilayer gels on a bio-mimicking tongue. Food Oral Processing Conference 2021. July 12th - 14th, 2021- Online. [Poster]

6. Potential of a newly developed biomimetic setup to better integrate the kinematic, mechanical and surface characteristics of tongue in oral tribological studies of foods. Food Oral Processing Conference 2021. July 12th - 14th, 2021- Online. [Poster]

# Research Exchange

At Wageningen University (The Netherlands) under the guidance of Dr. Markus Stieger and Dr. Elke Scholten. [April 2019 – May 2019]

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# Table of contents

# I
# Introduction

The quest to develop technologies to better understand and model consumer's sensorial perception of food is of paramount interest to meet the challenges of today's food transitions. The insights brought through these novel technologies in turn help in formulating the food for different sections of populations, especially ones with specific needs (from infants to the elderlies). Understanding the perception of texture with respect to food oral processing has thus gained a lot of interest in the scientific community. The reason behind this is the growing interest towards adding value to raw products through food processing, including efforts taken by food developers to create convenient and healthy products (with less fat, sugar etc.) or completely new products like plant based or cellular meat. The texture is also modulated to meet the specific needs of people suffering from physiological disorders like dysphagia etc. Unlike taste perception where there are specific receptors and most of the compounds responsible for certain perceptions are well catalogued, texture perception is much more complex due to its dynamic and multi-sensory nature. The sensory evaluation of food texture begins long before the food is put in the mouth. Its visual appearance, sound and tactile sensations when handled provide information about the mechanical properties of the food. However, it is clear that the oral phase plays a singular and dominant role in the sensory perception of texture, and thus in the appreciation or rejection of the food by consumers. While being broken down in the oral cavity to be made suitable for swallowing, food interacts with oral surfaces, leading to the stimulation of mechanoreceptors whose signals play an important role in the brain's processing of texture perception.

The efforts in characterizing food texture have evolved over the span of time. Mechanical characterization of food (whether bulk or interfacial properties) is possible through various instruments. However, these mechanical measurements sometimes do not precisely reflect the perceptions of texture perceived by a sensory panelist or by a regular consumer. There is some consensus in the literature is that such discrepancies in the correlation of the physical measurements and sensory data can be due to the fact that instrumental techniques (i) do not sufficiently take into account the oral physiology (oral surface, oral motions and biomechanics) and (ii) are mostly focused on specific individual stages of a food oral processing (ex. penetration test for chewing) and not on the dynamic process as a whole.

Hence the question that arises and which very much underscores the aim for this thesis is: *Is it possible to integrate these complexities of oral physiology in the in vitro instrumental methods, either by adapting existing setups to the need or by developing new ones?* This doctoral project was therefore aspired to understand the biomechanics of the tongue-food interface by designing, on the one hand, *in vitro* oral processing systems that take into account oral physiological variables; and on the other hand, developing original methods such as ultrasound-based techniques that are non-destructive, non-invasive and which can provide real-time analysis of the tongue-food-palate system. This work involved a multidisciplinary approach that required expertise in biomechanics, acoustics, and food science.

The PhD work is presented in the different chapters of this thesis manuscript. To begin with state of the art (**chapter 2**), which provides an important overview of the literature. This chapter aims at providing insights about food oral processing and its associated biomechanics, as well as the recent advancement in characterizing perception of texture through instrumental and sensorial methods. The chapter concludes by opening up the objectives of this PhD based on the identified research gaps in the literature.

**Chapter 3** is devoted to the first stepping stone of our approach where a novel rough and deformable tongue model was introduced to understand the mechanical breakdown of food gels under uniaxial compression. This chapter discusses in detail how the development of an ultrasound-based monitoring method can help to characterize the tongue-food mechanical interactions under the influence of imposed compression. The content of this chapter is peer reviewed and was published in the journal Innovative Food Science and Emerging Technologies.

Moving ahead in **chapter 4**, the use of the ultrasound method developed in the previous chapter was further explored to understand the deformation undergone by each individual entity of composite bilayer gels when compressed over a biomimetic tongue in an attempt to understand the perception of texture contrast. The ultrasound method brings novel insights in this field, as it allows to quantify the deformations of each structure, which is not possible by classical compression traction instruments like texture analyzer.  This work was done during my research exchange at the Wageningen University under the guidance of Dr. Makrus Stieger and Dr. Elke Scholten. The content of this chapter is submitted for publication in the peer reviewed journal Current Research in Food Science.

The next step towards the goal of achieving more realistic biomimetic setup was to develop a new prototype which can perform more complex motions (tongue-palate shearing) in addition to uniaxial compressions. The **chapter 5a** describes all the steps we have taken in order to establish a specification and to design a new biomimetic system from scratch. Once the setup was built, the first proof of concept study (**chapter 5b**) was conducted to characterize tribological phenomena with respect to tongue and food properties, as well as operating parameters like normal stress and shearing velocity. The content of this chapter was published in the peer reviewed journal Food Hydrocolloids.

Finally, the advancement that this thesis work brings in the field of food oral processing by developing biomimetic setups and analysis of the tongue food interface (both via ultrasound and force variations) are discussed with some complementary sensory and instrumental results in the general discussion part of the thesis (**chapter 6).**

# II
# State of the art

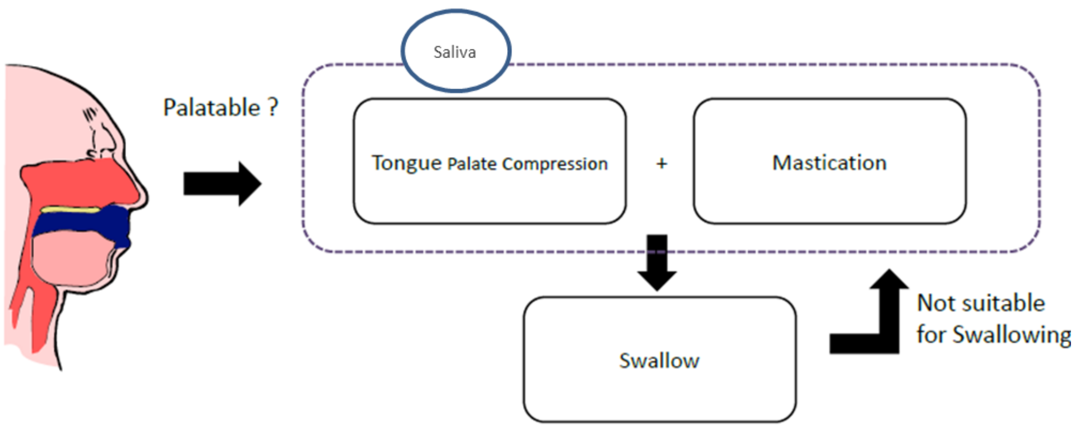
# Preface

*This chapter deals with in-depth literature review of different topics which were essential for completion of this doctoral thesis. The chapter is organized to discuss the food oral processing and texture perception, as well as their association with oral anatomy and physiology. The chapter also reviews the sensory and instrumental methods used for texture characterization and the challenges in correlating the physical measurements with sensory ratings. In particular, the review discusses novel approaches consisting in developing new quantitative ultrasound methods all by getting closer to realistic conditions, and consequently opening up the research gap and opportunity for this thesis. In conclusion, the chapter discusses the objectives of the doctoral work.*

## 2.1. Food oral processing

Oral processing is an important physiological phenomenon involved in food consumption. Once in the mouth, food is progressively reduced into particles by the effect of chewing by teeth and of shearing between the tongue and the palate. These particles are hydrated by saliva until they form a cohesive food bolus (Szczesniak, 2002). Food is thus gradually transformed into a form that must be swallowed comfortably (Chen, 2009).

Processing of food in the oral cavity can be divided into different stages, including ingestion, mastication, tongue-palate shearing and swallowing (Heath & Lucas, 1987; Chen, 2009). Food oral processing begins with either first bite or simply by the deposition of food on tongue surface. Once in the buccal cavity, food is further broken down by the teeth through the so-called phenomena of chewing or mastication. The chewing of food depends on the mechanical properties of food (Lucas, Prinz, Agrawal, & Bruce, 2002) and is often extended for better saliva incorporation, making it suitable for tongue-palate shearing. These stages of oral processing occur simultaneously, for an optimized management of the bolus. They all have an effect on sensory perceptions such as taste, aroma or texture, but also on the kinetics of nutrient release during digestion. Once the food bolus becomes suitable (safe enough to avoid risks of choking), swallowing is triggered. The residual food undergoes further tongue-palate shearing and is responsible for persistent mouth-feel. The figure (Figure 2.1) below presents a schematic description of the different stages of oral processing.




**<u>Figure 2.1:</u> Schematic representation of food oral processing.**

There are different factors (related to oral physiology and food properties) that can have an impact on how the food is orally processed. Therefore, it becomes imperative to first understand the oral anatomy and physiology and the associated biomechanics.

## Oral cavity

The oral cavity is the first part of the human digestive system, comprising of mouth (anterior portion guarded by lips) which opens into a cavity bearing jaws, teeth and tongue. The oral cavity is laterally supported by cheeks and the roof of the cavity is termed as palate, which is hard on the anterior part, but soft on the posterior part leading to the pharynx. The oral cavity size differs greatly from one individual to another and is important to estimate the total surface area for food-saliva interaction, as well the bite size. The estimation of the volume of oral cavity can simply be done by measuring the mouthful water. Moreover, acoustic techniques are also available like acoustic pharyngometer, allowing to measure a volunteer's airway size by graphically displaying the relationship between the cross-sectional area of the airway and distance down the airway in centimeters. Xue & Hao (2006) reported orders of magnitude of the volume of oral cavity by acoustic pharyngometry, within the range of 30–45 $cm^3$.

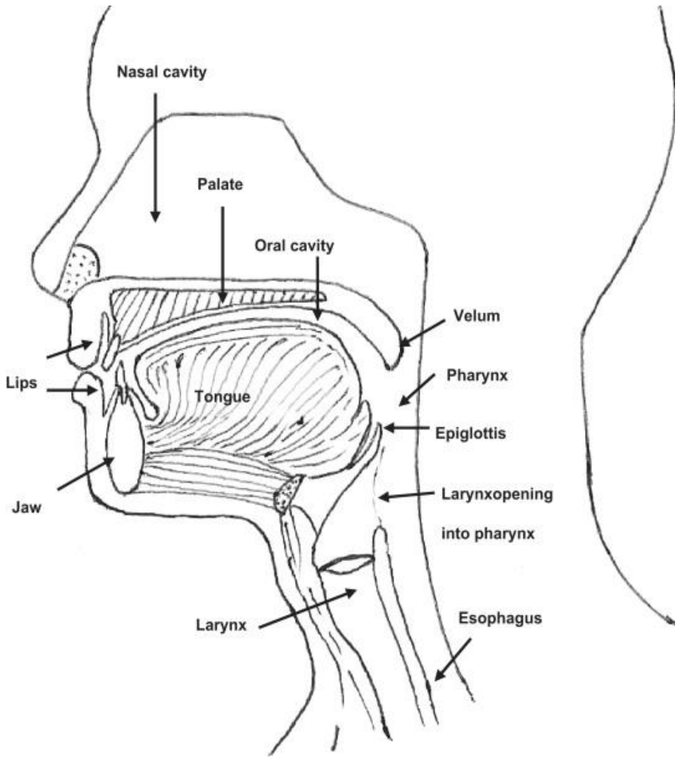



**Figure 2.2: Oral anatomy (Chen, 2009).**

## Jaws and teeth

The oral cavity is comprised of two sets of jaws: upper (maxilla) and lower (mandible). Lower jaw is connected to the skull with help of two joints called temporomandibular joints (TMJ). The jaws provide anchorage to teeth and facilitate occlusion (contact between upper and lower sets of teeth). The muscles like temporalis, masseter, medial and lateral pterygoids help in the movement of the mandible.

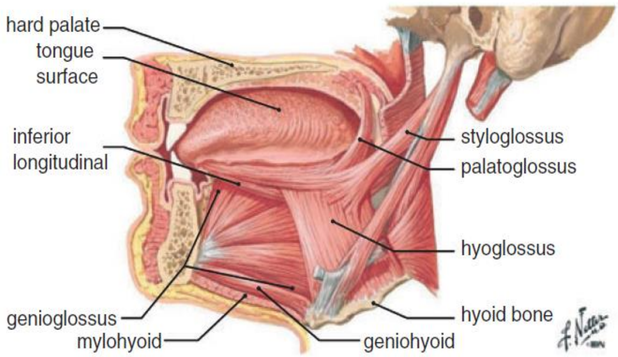
Teeth are hard bony structures covered with enamel. A normal adult has usually thirty two teeth, which are used for chewing food and making it suitable for swallowing. Teeth are characterized in three main categories based on their function: incisors to cut, canines to cut and tear, and finally molars to grind the food during chewing. Beyond their role in mechanical breakdown of food, the teeth are also sensitive to the force applied to them thanks to periodontal ligament, and can sense the particle size of food (Koc, Vinyard, Essick, & Foegeding, 2013). During the oral processing of food, an important variability between individuals was noticed in jaw muscle activity (Kakizaki, Uchida, Yamamura, & Yamada, 2002; Pereira, Duarte Gaviao, & Van Der Bilt, 2006), mandibular jaw movement (K. Hiiemae et al., 1996; Peyron, Mioche, Renon, & Abouelkaram, 1996), masticatory forces (Mioche & Peyron, 1995; Periera et al., 2006), chewing cycle duration (Periera et al., 2006; Gavião, Engelen, & Van Der Bilt, 2004; Jalabert-Malbos, Mishellany-Dutour, Woda, & Peyron, 2007) and chewing rate (Brown, 1997).

**Tongue**

The tongue is a muscular organ present in the oral cavity which is crucial for oral processing and sensorial perception of food. The tongue is capable of sensing texture, taste and temperature. It facilitates the manipulation of food and of the bolus by moving it into the cavity, mixing it with saliva, shearing it against the hard palate and initiating swallowing (K. M. Hiiemae & Palmer, 2003).

<u>Tongue anatomy</u>

The tongue is composed of eight muscles (Figure 2.3), divided into two classes: extrinsic and intrinsic. The extrinsic muscles connects the tongue body to the bone while the intrinsic muscles are present inside the tongue (Gaige, Benner, Wang, Wedeen, & Gilbert, 2007; Shawker, Sonies, & Stone, 1984). The extrinsic muscles are responsible for the movement of the tongue: the genioglossus which arises from mandible (allows the protrusion of the tongue), the hyoglossus which arises from hyoid bone (is responsible for retraction and depression of the tongue), the styloglossus which arises from styloid process of the temporal bone (draws the side of the tongue to create channel for swallowing), and the palatoglossus which arises from the palatine aponeurosis (facilitates tongue-palate contact). The intrinsic muscles for their part aid in changing the shape of the tongue: lengthening and shortening of the length, and curving the surface (Chen, 2009).



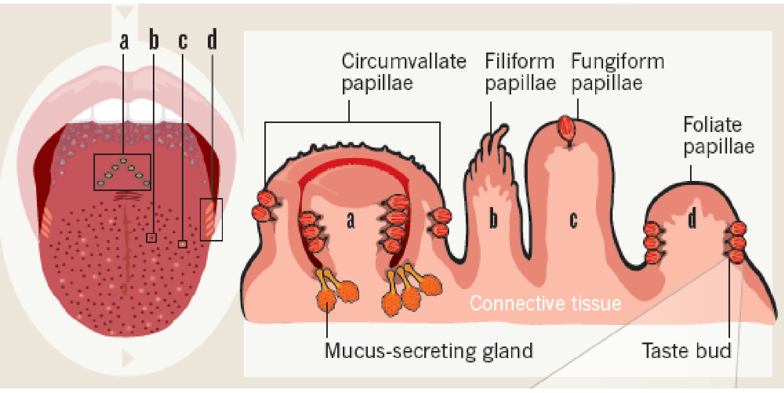

**<u>Figure 2.3:</u> Longitudinal section of tongue.**

<u>Tongue mechanical properties</u>

The human tongue is also a complex biological tissue from a mechanical perspective. The tongue is termed as a muscular hydrostat, which means that its volume remains constant when it is contracted, or when in motion (Napadow, Chen, Wedeen, & Gilbert, 1999). The rigidity of the tongue was measured by various techniques: mechanical indentation, mechanical compression and magnetic resonance elastography (Cheng, Gandevia, Green, Sinkus, & Bilston, 2011; Gerard, Ohayon, Luboz, Perrier, & Payan, 2005; Ishihara et al., 2013). The rigidity of the tongue, however, changes significantly between the "at rest" state ($E = 12.2 \pm 4.2$ kPa) and the "contracted" state ($E = 122.5 \pm 58.5$ kPa) (Ishihara et al., 2013). Moreover, tongue mechanical properties have been shown to vary greatly among healthy individuals (17-75 kPa), and then to correlate with the threshold for tongue-only oral processing of food (Alsanei, Chen, & Ding, 2015).

<u>Tongue surface properties</u>

The surface topology is another peculiarity of this organ, as the dorsal surface of the tongue bears numerous tiny protrusions called papillae. These papillae have different shapes and sizes, and are randomly distributed over the surface. They help in perception of taste as they are equipped with different taste receptors. Having brush like structure, the tongue papillae are also involved in tactile perception, as they interact with food and stimulate the mechanoreceptors present underneath. There are four types (Figure 2.4) of papillae on the surface of the tongue: filiform, fungiform, folate, and circumvallate (Jung, Akita, & Kim, 2004; Ranc, Servais, Chauvy, Debaud, & Mischler, 2006). The filiform papillae are present at high densities across the tongue's surface, greatly contributing to its overall roughness. In past, researchers have tried to characterize the surface profile of tongue, however, it's not easy mostly for technical reasons as it is almost impossible to do profilometry on humans. Hence there is requirement of a sophisticated molding methods instead. However, it is never easy to ensure that casts are fully representative of the actual topology of the tongue surface.




**<u>Figure 2.4:</u> Different types of papillae on present on the tongue surface (Trivedy, 2012).**

The roughness of a surface is characterized in particular by a parameter called Ra (arithmetic average height), which can be defined as the average absolute deviations of the roughness irregularities from the mean line over one sampling length. Studies have reported the order of magnitude of Ra for human tongue: Nagaoka et al. (2001) published it between 13.5 and 108.4 µm, Ranc, Servais, Chauvy, Debaud, & Mischler (2006) found it between 10 to 100 µm, and Uemori, Kakinoki, Karaki, & Kakigawa (2012) estimated it between 42.5 to 101.4 µm. More recently, Andablo-Reyes et al. (2020) published dimensions of fungiform and filiform papillae by using 3D printing technology for development of artificial tongues with negative molding methods.

**Table 2.1: Dimensions and density of fungiform and filiform papillae present on human tongue (Andablo-Reyes et al., 2020)**

| | Papillae density (per $10^{-4}$ $m^2$) | | Papillae dimensions | | | |
|---|---|---|---|---|---|---|
| | | | Fungiform | | Filiform | |
| | Fungiform | Filiform | Diameter (µm) | Height (µm) | Diameter (µm) | Height (µm) |
| Mean | 13.5 | 160 | 878 | 390 | 355 | 195 |
| Deviation | 1.5 | 30 | 97 | 72 | 40 | 6.5 |

The tongue surface topology also impacts wettability properties. The work of Ranc et al. (2006) showed that the surface of the pig's tongue has different wettability depending on whether it was coated with saliva (contact angle with water 50.5 ± 2.4 °) or not (contact angle with water 77.3 ± 4.1°).

Tongue movements

Also, the movement of the tongue varies greatly depending upon the properties food, especially during oral processing. (Peng & Miethke, 2000) found that the speed of the tongue during processing of liquid food ranges between 2 $mm.s^{-1}$ to 32 $mm.s^{-1}$. Hiiemae & Palmer (1999) proposed a model explaining intra-oral events associated with the management of solid foods varying in textural properties. The model encompasses two major stages of food transport and associated tongue movements. The first stage (Stage I) is when a pull-back tongue motion makes it possible to place the ingested food where it will undergo the mechanical breakdown (whether by mastication or tongue-palate compression). After this stage, the tongue facilitates the incorporation of saliva with food particles, guides food to the different types of teeth, and helps bringing all the hydrated particles together to form a cohesive bolus. At the end of oral processing, Stage II transport helps in the progressive transport of processed food to the oropharyngeal part of oral cavity, just before the initiation of the peristalsis that commences swallowing. Throughout these stages, numerous biomechanical phenomena occur. They involve the properties of the food which evolve throughout oral processing. But these phenomena also include the very important contribution of the intrinsic properties of the tongue. It is thus clear that whatever the type of food and whatever the stage of oral processing, the tongue has a very important role to play**.**

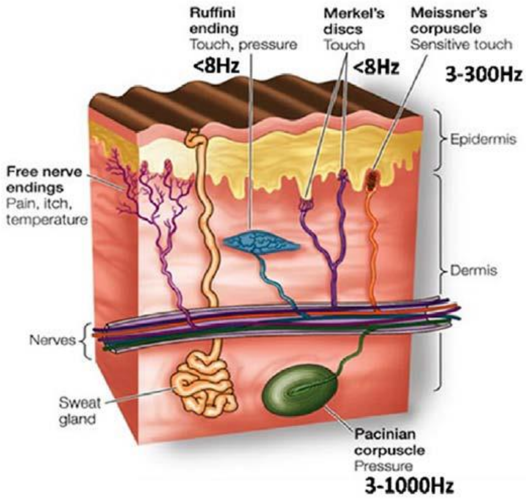
Tongue's role in oral strategy management

Sensory feedback and oral strategy management is an important aspect in food oral processing. Food oral management is characterized by a series of choices of successive oral operations carried out in the right order and in good coordination. It is important to underline that the tongue has a central role in these choices and follow-up of the strategy of food oral management (Heath, 2002). It ensures that the food follows a process throughout which the sensory appreciation is guaranteed, and at the end of which safe swallowing can be triggered. For this reason, in addition to motor nerves, the tongue is also equipped with sensory nerves that allow for the translation of different sensory modalities, like, nociception, thermoception and chemoception and mechanoception. The conversion of sensory stimuli into nerve signals then is processed by the brain to adapt tongue movements for every particular need.

For questions related to the biomechanics of the tongue and its involvement in texture perception, it is intuitive to focus on the links between tongue movements and tactile perceptions resulting from the stimulation of the mechanoreceptors. As we know, tongue is able to sense the texture of food as it bears many mechanoreceptors ((Foegeding, Vinyard, Essick, Guest, & Campbell, 2015). Mechanical stimuli (in the form of stresses and strains due to various mechanical interactions between tongue tissue, food and saliva) result in the stimulation of the mechanorecptors, which then translate them into neurotic signals which are processed by the brain as the texture attributes of the food. Precisely, when food comes into contact with tongue surface, stresses and deformations are produced and reach the receptive fields of the mechanoreceptors (which vary in size and depth). The topological profile of the tongue surface can thus modulate the transmission of mechanical stresses and hence the tactile stimuli generated.

There are two classes of mechanoreceptors located at different depths within the tissues (like skin, Figure 2.5): slow adapting and rapid adapting (Foegeding et al., 2015). However, the studies on tongue mechanoreceptors are sparse.

Three out of the four types of low-threshold mechanoreceptors present in cutaneous tissues have been found to be present in tongue tissue : SA1, SA2 and RA1 (Drew, Rugiero, & Wood, 2007; Trulsson & Essick, 1997). The Slowly Adapting type 1 (SA1) mechanoreceptors, whose endings- are called Merkel's disks, produce sustained responses to static stimulation. Slowly Adapting type 2 (SA2) mechanoreceptors, with Ruffini's corpuscles, also lead to responses proportional to the pressure applied, but deeper and with larger receptive fields than SA1.




**<u>Figure 2.5:</u> Types of mechanoreceptors in the skin (Cesini et al., 2018).**

The Rapidly Adapting type 1 (RA1), with Meissner's corpuscles, are shallowly located, have small receptive fields, and produce transient responses to the sudden onset and offset of tactile stimulations. The Rapidly Adapting type 2 (RA2, Pacinian corpuscles), which are sensitive to higher vibration frequencies than RA1, have not been reported in human tongue tissues so far. The oral region is less sensitive to vibrations and mechanical transients than the human hand (Essick & Trulsson, 2009). Highest perceived frequencies are of the order of 400Hz (Upadhyay & Chen, 2019).

**Salivary glands and saliva**

Saliva is a colorless bodily fluid (mainly water - 98 – 99.5 %; pH 5.6 – 7.6) secreted by three pairs of salivary glands in the oral cavity. The three types of glands are: the sublingual glands (present underneath the tongue), submandibular glands (present underneath the jaw) and parotid glands (present underneath the ear). The secretion of saliva in the oral cavity is stimulated by different stimuli: visual, olfactory, gustatory and mechanical (act of chewing). The salivary flow varies greatly from one individual to other, in average between 0.77 and 4.15 ml/min (Chen, 2009) (stimulated saliva flow rate is measured during chewing, a small parafilm sheet can be used for this purpose).

It is composed of electrolytes (P, $Na^+$, $Ca^{2+}$, $Cl^-$, $Mg^{2+}$), as well as proteins (mucins and enzymes like alpha-amylase, immunoglobulins, glycoproteins) (De Almeida et al., 2008). It lubricates the oral cavity, initiates the digestion process (with α-amylase), and plays an important role in taste perception (release of aroma and taste compounds) and texture (material continuity between food and mucosa) (Stokes, Boehm, & Baier, 2013).

Saliva has an important role in the oral processing. It assists in deconstruction of the food by providing hydration and lubrication. A solid product requires a long mastication and saliva incorporation before being reduced sufficiently to be swallowed, whereas a liquid product remains in the mouth for only a short time before swallowing and does not require deformation by action of the organs of the oral cavity. Hutchings and Lillford (1988) presented a model explaining oral retention time of different foods with respect to degree of structure and degree of lubrication required (Figure 2.6). The model concludes that higher is the degree of structure more time is retained in the mouth for size reduction and saliva incorporation.

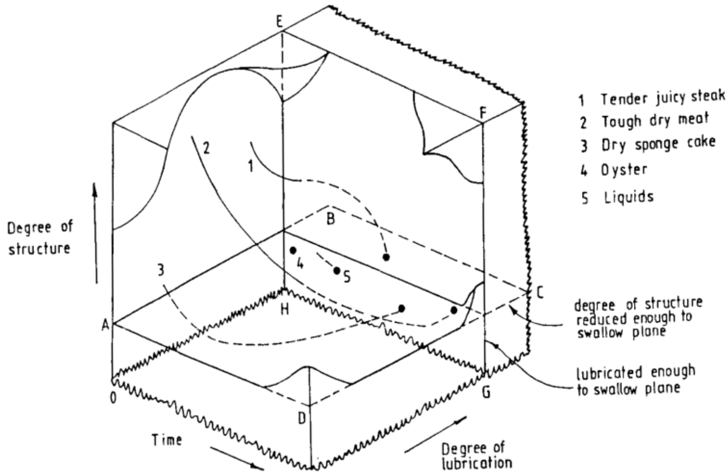



**Figure 2.6: Food transformation during oral processing (Hutchings and Lillford, 1988).**

Drago et al. (2011) investigated the role of saliva pH, rate of incorporation and spreading ability on bolus rheological properties (8 different dairy products). The authors also pointed out some correlations between lysozyme activity and bolus properties such as variation of pH, hydration by saliva, higher rheological properties etc.

According to Stokes et al (2013), the amphiphilic proteins contained in saliva, the mucins, adhere to surfaces and by hydrogen bonding, create interactions between the food and the walls of the oral cavity and ultimately with receptors. The lubricity of the saliva is due the mucin protein and has been studied widely in literature as how it facilitates mitigation of oral friction and how its precipitation by certain compounds like polyphenols or tannins could lead to the perception of astringency (Gibbins & Carpenter, 2013; C. A. Lee, Ismail, & Vickers, 2012; Ployon et al., 2018).

## 2.2. Food texture perception

The definition of the term "texture" in the food industry has evolved considerably over time. A consensus was finally found around the definition given by Szczesniak in 1998: "Texture is the sensory and functional manifestation of the structural and mechanical properties of food, detected by the senses of vision, hearing, touch and kinesthetic perceptions". Texture depends on multiple parameters and has many characteristics; it is correlated to the structure of the food (at the molecular, microscopic and macroscopic scales); it is a synthesis of information from different senses. Although it is partly apprehended by sight or touch before the food is introduced into the mouth, the oral phase plays a fundamental role in texture assessment (Szczesniak, 2002; Wilkinson, Dijksterhuis, & Minekus, 2000). In particular, the contribution of the tongue and its multiple mechanoreceptors is essential.

As mentioned above, texture is very much required for the overall appreciation of the food. Manipulating texture and creating contrasts when formulating foods impacts the sensory experience of the consumer (Palczak, Giboreau, Rogeaux, & Delarue, 2020; Szczesniak & Kahn, 1984). Texture perception often occurs simultaneously with other types of perceptions like taste and aroma, and the interactions between them are also scientifically reported (Saint-Eve et al., 2011). Texture is also important in the identification (and subconscious memory) of a food product. It was thus shown that when food is blended before eating, only 40 % percent of the studied products were recognized correctly (Schiffman, 1977). In addition to sensory appreciation, texture is also important for safe consumption of food especially, for infants, elderlies and people with eating and swallowing disorders.

The vocabulary used to describe texture perception by people is wide (hard, soft, brittle, dry, moist etc.). Often these terminologies can be abstract. The choice of words to describe one particular attribute of texture is governed either by food’s mechanical properties or can also be by the interaction of oral surface with the food. As a consequence, the magnitude of these qualitative attributes needs to be translated into measurable mechanical properties such as Young’s modulus or friction coefficient (discussed in the next section dealing with texture characterization).

## 2.3. Food texture characterization

### 2.3.1. Sensory Methods

As mentioned before, texture perception is a sensory property which can only be described in a subjective way by the people. In sensory science, different methodologies are used to comprehend the perception of different textural attributes with trained or untrained panelists. Some methods allow to describe the perceptions of texture globally like Qualitative Descriptive Analysis (QDA) and texture profile method. Other methods are temporal, they are appropriate for assessing sensory attributes as a function of oral processing time. Dynamic texture assessment can be done by methods like: Time Intensity (TI), Temporal Dominance of Sensations (TDS). These methods are complementary to each other when evaluating the texture of a food. Their association allows to obtain a quantitative and temporal description of it.

Descriptive methods

To begin with, the QDA method was conventionally developed for assessing the flavor profile or appearance of products, and it can be adapted for texture analysis with related terminologies. The principle is to use the capacities of a subject to verbalize his perceptions. The sensory evaluation works with a group of individuals called a panel. During training, the panel generates a list of sensory attributes. It describes the nature of the perceptions and quantifies their intensity, in order to give an accurate, reproducible and understandable product identity card. Descriptive profiles involve the evaluation of products in repeated tests to obtain a complete quantitative description. The performance of the panel is considered as successful when it meets the following three criteria: (i) homogeneity (agreement among judges), (ii) discriminative power (the ability to differentiate products), and (iii) repeatability (across judge and panel).

The perception of texture perception changes at different stages of oral processing, a method which takes in account this peculiarity is needed.

Brand, Skinner & Coleman (1962) for the first time proposed a method exclusive for texture analysis, and named it texture profile analysis. In this method the terms for describing texture were grouped according to their common physical state like mechanical (hard, soft, spongy...), geometry (shape, size…) and other specificities like moistness or fat perceptions. These attributes can then be rated on intensity scales at different stages of oral processing like first bite, chew down and after swallowing.

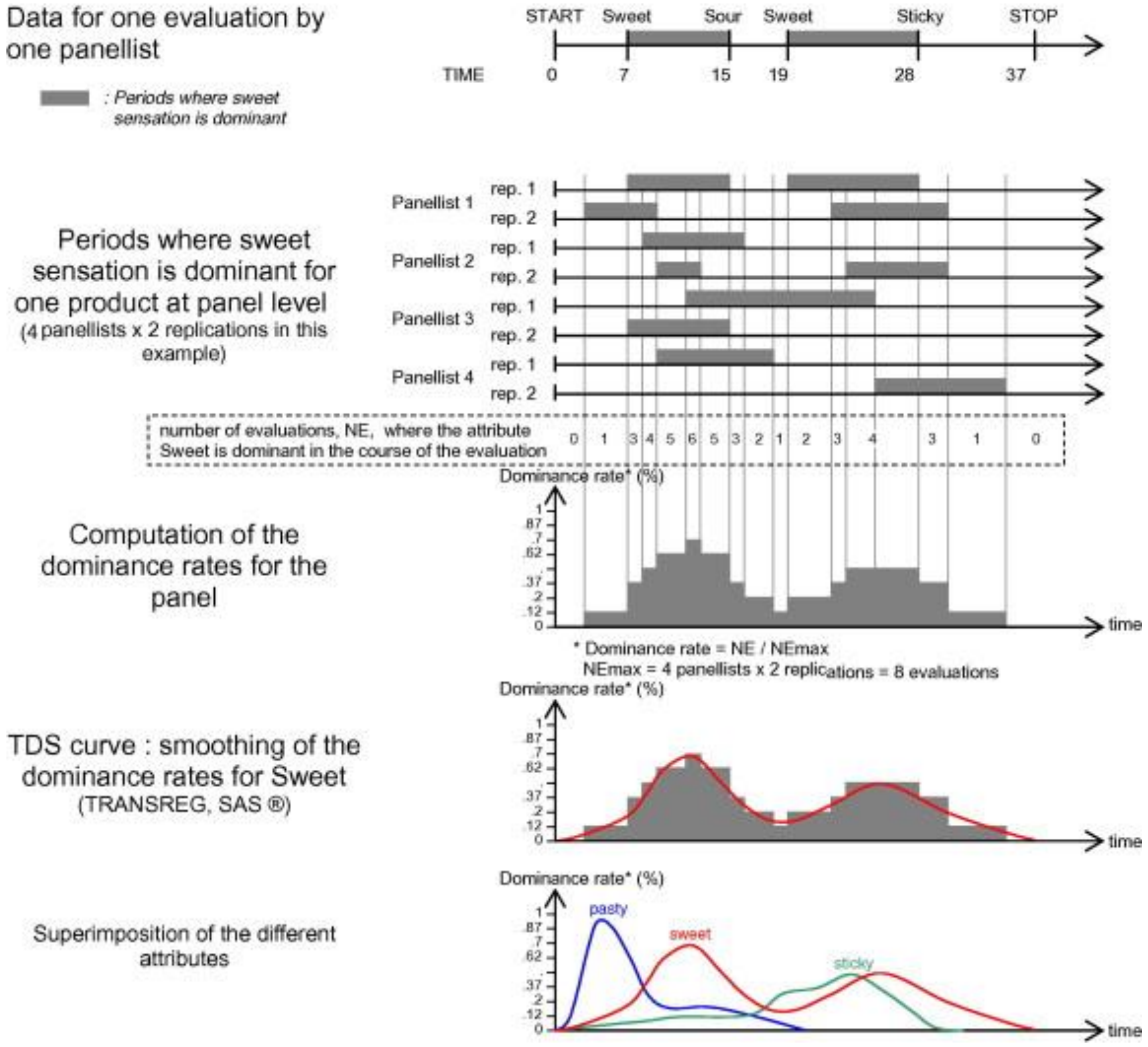
<u>Dynamic Methods (Temporal methods)</u>

Dynamic or temporal methods have the advantage over descriptive methods by allowing the evaluation of texture perceptions as a function of time during the oral processing. Unlike descriptive methods which provide sets of pre-decided instructions to the panelist about time of tasting, temporal methods let the panelist rate a particular attribute in a continuous manner.

Time Intensity (TI) analysis is one such method often used to analyze the evolution of the intensity of one or two attributes over time during the entire oral processing. The method requires attentiveness and reactivity, which limits the number of attributes to be tested simultaneously. This makes this method time consuming and expensive if several products have to be tested on various textural attributes.

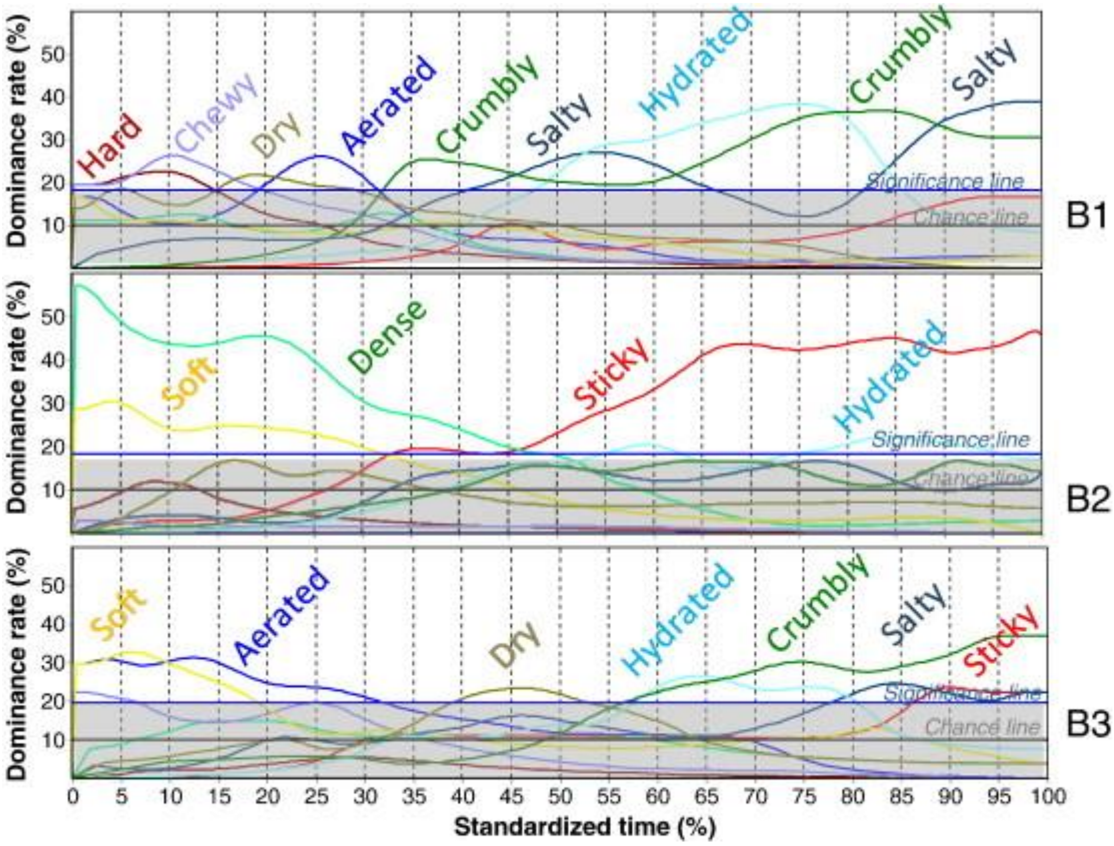
Time dominance of sensation method on the other hand is comparatively recent and provides much wider scope than TI (Pineau et al., 2009). TDS method is used to track the dominant sensation over time, by citing the attributes that attract the most attention during the consumption of a product. The advantage of this method over TI is that it requires very little training of the panelists and multiple attributes (up to 10) can be analyzed in one go. However, it is important to keep in mind that TDS method is not used to rate intensity of the perception of a certain attribute but the frequency with which it comes to the attention of the panel members during the consumption. The choice of the attributes for the TDS method is very crucial as it impacts the outcome of the sensory study.

Pineau et al., 2009, when presenting the method first time, explained well the methodology to compute TDS curves (Figure 2.7). The procedure takes into each attribute separately. For each temporal point, "the proportion of runs (subject × replication) for which the given attribute was assessed as dominant is computed. These proportions, smoothed using the TRANSREG procedure of SAS®, are plotted against time and called TDS curves" (Pineau et al., 2009). (TRANSREG procedure uses transformation which is a non-iterative smoothing spline)




**<u>Figure 2.7:</u> Steps involved in evaluation of data obtained from TDS (Pineau et al., 2009).**

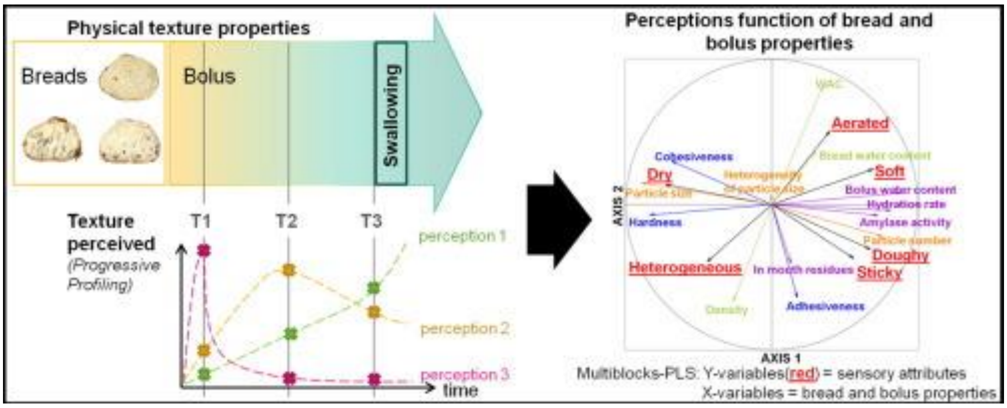
A study performed by Panouillé, Saint-Eve, Déléris, Le Bleis, & Souchon (2014) could be presented as one example out of numerous studies that have used TDS method for analyzing the dynamics of texture perceptions. In this aforesaid article, temporal dominance of texture attributes were studied for different breads (varying in fat and sugar content). It can be observed from the figure 2.8, three breads (B1, B2 and B3) had a different perception of texture at the beginning of the consumption. All breads were perceived as hydrated when approaching the end of consumption. Also when comparing the TDS curves of the three breads one can notice the appearance of salty perception at different parts of consumption. These observations obtained from TDS method were undoubtedly very interesting, and proves the efficacy of this method especially for texture studies.




**<u>Figure 2.8:</u> An example of TDS obtained for three breads samples (Panouillé et al., 2014).**

It is very evident that the TDS has evolved as one of the most suitable method in following the dynamics of texture perception. However, the inability to rate the intensity of the textural attribute is one drawback.

Progressive profiling is another temporal method which provides opportunity of rating the intensity at different time intervals during processing (unlike TDS where the dominance rating is almost continuous). This method was first used to comprehend texture perceptions in cheddar cheese (Jack, Piggott, & Paterson, 1994). In progressive profiling, the trained panelists are instructed to rate different attributes on a line scale at a different predetermined time window. In progressive profiling, many attributes can be rated in one evaluation. Jourdren et al. (2016) used this method for comprehending the impact of bread structure on the food oral processing and texture perception. The data obtained from the progressive profiling method is very exhaustive and can be used to develop different statistical models as done in the said publication (Figure 2.9).




**<u>Figure 2.9:</u> Use of Progressive profiling for studying the impact of bread structure on texture perception (Jourdren et al., 2016).**

## 2.3.2. Instrumental Methods

As discussed previously for the sensorial methods, the texture attributes are subjective and sometimes could be abstract, in the sense that these attributes do not necessarily provide a clear understanding of the physical and mechanical phenomena behind them. Sensorial methods are also time consuming and expensive. Therefore the need for instrumental analysis of food (or bolus) in order to comprehend the sensory perception based on its mechanical and structural properties becomes evident. The characterization of the mechanical properties of food can be done at two levels, including (i) the bulk properties of the food and of the bolus (shear viscosity, yield stresses and moduli), and (ii) the properties resulting from the interactions between the food and oral tissues (like friction).

The correlation between instrumental characterizations and sensory analysis data could be complimentary, and should be encouraged for the understanding of the mechanical aspects of food breakdown with resultant textural perception. This point and associated challenges are further discussed in the next section.

### <u>2.3.2.1 Bulk Rheology</u>

Rheological methods are among the first instrumental methods which have been used to study the physical mechanisms behind texture perceptions. Rheology allows to link force (or stress) to the deformation of materials. Therefore, it is suitable to describe the breakdown of food to mechanical stresses encountered during oral processing. With time, classical rheological methods along with other applied methods have been developed to take into account the experimental challenges related to food bolus (Stokes et al., 2013).

Mechanical characterization of solid foods

For instrumental characterization of the texture of solid or semi-solid foods, compression tests could be carried out. Using compression/traction equipments such as so-called Texture analyzers, food is deformed uniaxially between two rigid surfaces. A controlled force or displacement is applied to the product and its response in terms of force and deformation is measured as a function of time. This makes it possible to characterize different types of products and to analyze their mechanical properties. Single or double compression, penetration, spreading, back extrusion and tensile tests can be performed.

Single uniaxial compression is also very often used to measure fundamental mechanical parameters. The results of a uniaxial compression can be presented in the form of a stress-strain curve to determine the Young's modulus, yield strength, fracture point etc.

One of the most widely used compression tests to characterize texture is Texture Profile Analysis (TPA) (Szczesniak, 1963). This method aims to establish a direct link between mechanical and sensory properties of the product. The TPA protocol requires double compression of the food, to collect a curve tracing the evolution of the force over time (Figure 2.10). The double compression was chosen to mimic the action of the teeth during chewing or the action of the action of the tongue during tongue-palate compression. Friedman, Whitney, & Szczesniak (1963) illustrated a TPA curve and discussed the various resulting quantitative parameters related to food mechanical properties. In Figure (2.10), hardness was defined as the peak force during the first compression cycle. Adhesiveness on the other hand was associated with negative force area A3. And cohesiveness was defined as the ratio of positive force area during the second compression to that during the first compression (A2/A1).

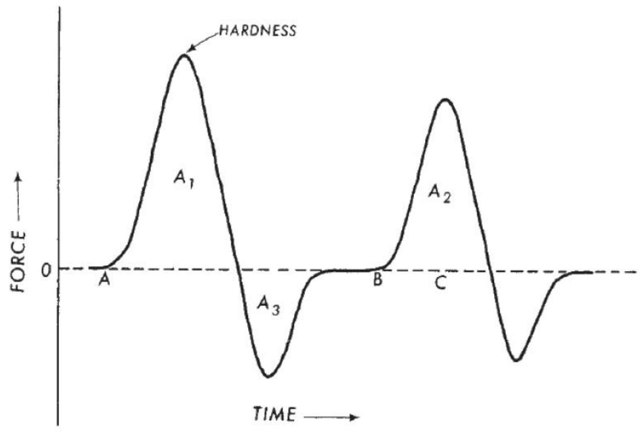



**Figure 2.10: A typical texturometer curve (Friedman et al, 1962)**

For liquid or semi-solid foods (which are not suitable for compression testing), shear measurements are preferred. The shear rotational tests allow to estimate the viscosity of the product, while oscillatory tests provide insights about its viscoelastic properties (Figure 2.11).

Viscosity is measured by applying a tangential stress to a fluid located between two solid surfaces with varied geometries such as coaxial cylinders; cone-and-plate; rotating disc; parallel plates.

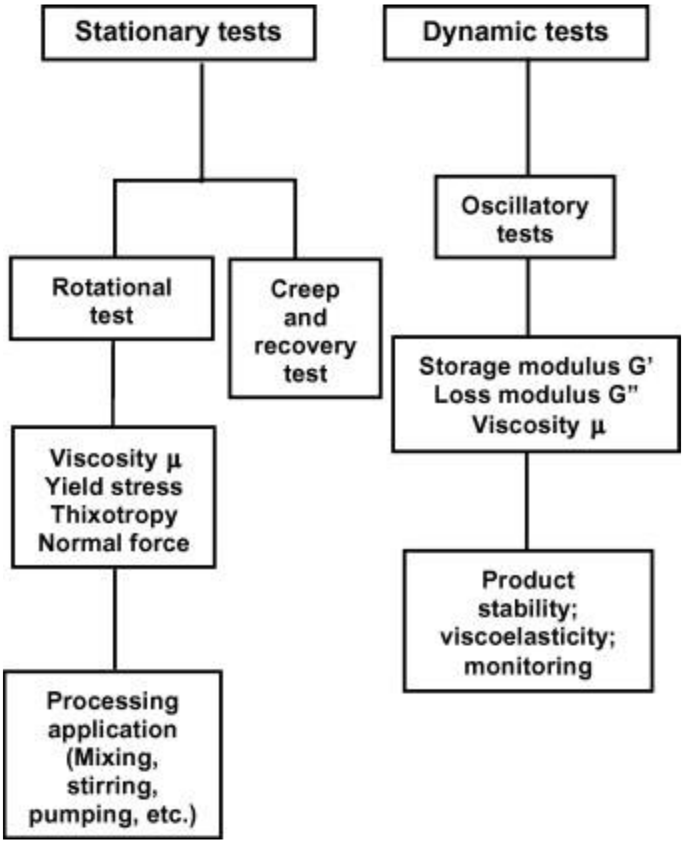
The shear stress ($\tau$) is defined as the ratio between the tangential force (F) measured between the solid surfaces and the area under the plate.

Then, the dynamic viscosity $\eta$ is the ratio between the stress and the velocity gradient $\dot{\gamma}$, also called shear rate (expressed in $s^{-1}$)) formed within the fluid:

$$\eta = \tau/(\dot{\gamma})$$

Where, $\eta$ is expressed in Pa.s, Poise or centi poise (1cP = $10^{-3}$ Pa.s).

The rheometers make it possible to impose and/or measure fundamental quantities such as shear rate $\dot{\gamma}$, shear stress $\tau(t)$ and viscosity $\eta$. This allows the derivation of flow and viscosity curves, which are characteristic of the product.




**<u>Figure 2.11:</u> Different rheological tests for food texture assessment (Tabilo-Munizaga et al., 2005).**

The flow curves represent the tangential stress versus velocity gradient ($\tau$ versus $\dot{\gamma}$). The viscosity curve represents the dynamic viscosity as a function of the velocity gradient ($\eta$ as a function of $\dot{\gamma}$). Figure 2.12 presents an example of variation of viscosity of different food products on application of shear stress.

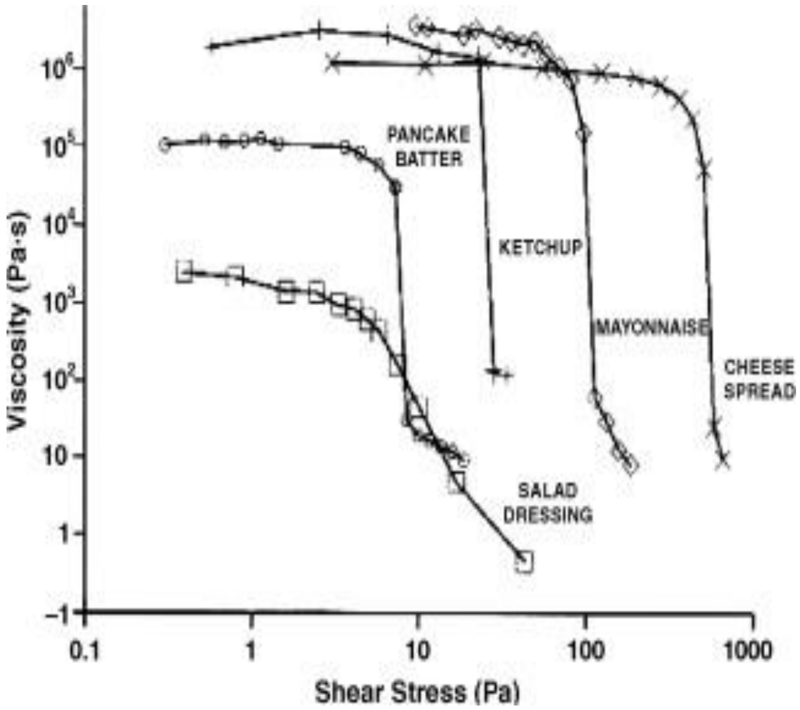



**Figure 2.12:** **Variation of viscosity with shear stress (Tabilo-Munizaga et al., 2005).**

The behavior of the fluid could be Newtonian or non-Newtonian. The viscosity of Newtonian fluids is independent of stress or shear rate. In contrast to Newtonian fluids, fluids with a non-constant velocity gradient behavior have a viscosity called "apparent viscosity". The non-Newtonian behavior can be further classified into (i) Shear thinning: when the viscosity decreases with applied shear (ii) Shear thickening: when the viscosity increases with applied shear. The shear thinning behavior is the most common in food as seen in figure 2.12. Many foods have rheological properties that depend on time and the thermomechanical history of the material. This is called thixotropy.

Research studies have also used sensory analysis methods (discussed previously) for complementing the instrumental measurements (Table 2.2):

**<u>Table 2.2:</u> Instrumental and sensory characterization methods used in tandem for texture analysis**

| Food product | Parameter | Instrumental method | Sensory method | Research reference |
|---|---|---|---|---|
| Breakfast cereals | Hardness, cohesiveness, springiness and adhesiveness | Texture profile analysis | TDS | Peyron et al., 2011 |
| Bread | Hardness, adhesiveness and cohesiveness | Texture profile analysis | TDS and PP | Jourdren at al., 2016 and 2017 |
| Emulsion filled gels | Peak force | Penetration test | TDS, PP and QDA | Devezeaux de Lavergne et al., 2015 and 2016 |
| Cheese | Spreadability | Spreading test | TDS, PP | Saint-Eve at al., 2015 |
| Biscuit | Flow behavior index | Back Extrusion | TDS | Young et al., 2013 |
| Custard | Apparent viscosity | Flow rheometry | QDA | De Wijk et al., 2006 |
| Custard | Storage modulus, loss modulus | Oscillatory rheometry | QDA | De Wijk et al., 2006 |
| Bread | Particle size | Image analysis | TDS, PP | Jourdren at al., 2016 and 2017 |
| Cornflakes | Peak force | Compression and acoustic response | QDA | Chaunier et al., 2005 |
| Sponge cakes | Apparent viscosity | Capillary rheometry | Oral comfort perception | Assad-Bustillos at al., 2019 and 2020 |

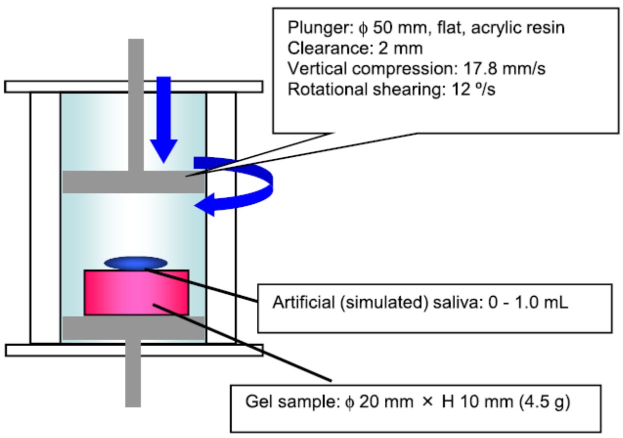
Although the classical instrumental methods have proven to be very useful in somehow establishing links between food mechanical properties and sensory perceptions, the scientific community gradually focused on developing methods which integrate the physiological and biomechanical characteristics of oral cavity.

<u>Novel measurements tailored for food oral processing</u>

- <u>Characterization of the bolus</u>

As food undergoes major transformations during the oral processing, one can assume that mechanical properties of the swallowed bolus would be quite different from the initial food ingested, and so will be the texture perception. Therefore, it becomes very necessary to characterize the rheological properties of bolus as well. This information can also be useful for understanding the rheological behavior of the bolus when swallowing and hence could bring key insights when designing products for populations with special needs, like for elderlies or for people with dysphagia.

As mastication during the processing of solid foods leads to fragmentation, important studies have been performed on natural products (peanuts, olives, chicken ham etc.) to understand the particle size distribution in the bolus and its impact on deglutition (Jalabert-Malbos, Mishellany-Dutour,Woda, & Peyron, 2007; Mishellany-Dutour, Woda, Labas, & Peyron, 2006; Peyron, Mishellany-Dutour, & Woda, 2004). These studies manly involved the subjects with strict dental criteria like complete dentition and no known masticatory disorder. Furthermore, towards more controlled in vitro approach, artificial mastication simulators have also been developed (Mielle et al., 2010; Salles et al., 2007; Woda et al., 2010).

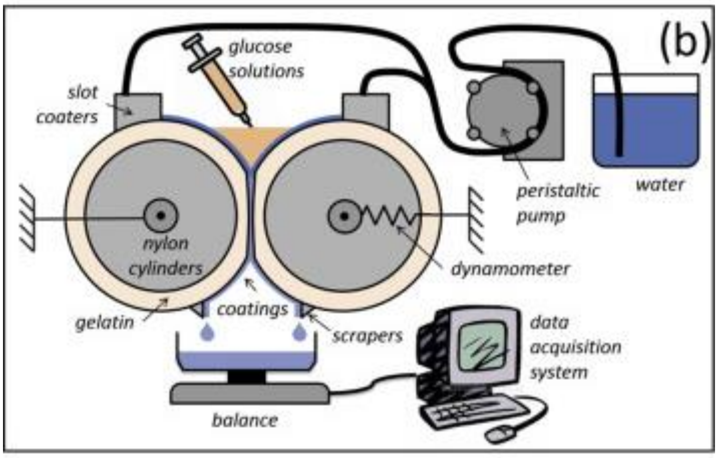



**<u>Figure 2.13:</u> Artificial mastication simulator for preparation of model bolus (Ishihara et al., 2011b).**

**Semi solid gels have also been used as model food** to understand the rheology of boli with viscoelastic properties. Ishihara et al. (2011a & b) developed a mechanical simulator to investigate the rheology of a bolus formed from instrumental mastication of gels in presence or absence of artificial saliva. The gels were compressed and sheared in a reciprocating manner using a flat plunger mimicking the action of a human jaw (Figure 2.13). The authors studied the frequency dependence of the viscoelastic properties of different gels (with homogeneous and heterogeneous structure). The role of saliva on the viscoelastic properties of gels was also explained with respect to the hydrophilic nature of the obtained bolus.

In the similar way, the rheological properties of the bolus and its interaction with saliva also becomes important during swallowing.  Number of studies have come up with custom-made setups and ranges of model foods to comprehend the subject better (Preciado-Méndez et al., 2017; Salinas-Vázquez et al. 2014).

Mathieu et al (2018) developed an in vitro elastohydrodynamic model (Figure 2.14) for in-line measurement of the thickness of the coatings resulting from the flow of bolus through the pharynx. The resultant coating on the pharyngeal mucosa were found to be dependent on the food properties (viscosity and density) as well as on physiological variables like lubrication by saliva, velocity of the peristalsis, and to a lesser extent, the deformability of the pharyngeal mucosa.




**<u>Figure 2.14:</u> In vitro simulator for pharyngeal swallowing phase (Mathieu et al., 2018).**

- <u>Food breakdown during tongue-palate compression</u>

Deformability of the tongue and its role in the breakdown of food has gained interest. Biomimetic tongues have been considered, made of different materials like silicone rubbers (Ishihara et al., 2013), acoustic gel pads (Gao et al., 2016) and urethane gels (Kohyama, Ishihara, Nakauma, & Funami, 2021). These methods have coupled classical compression-traction instruments with innovative and fit-for-purpose techniques like imaging and ultrasound elastography.

Figure 2.15 shows an illustration of this tendency with the work done by Ishihara et al. (2014). In this study, model gels (made from agar and gellan gum) were compressed over artificial tongues made of silicone rubber. The study could relate to the fracture of agar gels to that of their mechanical properties, when they were compressed over artificial tongues (Young's modulus: 55 kPa). As they could infer that the gels which fractured under 50% deformation were likely to be manipulated by tongue-palate compression while ones requiring greater deformation are broken down by the teeth. As a consequence it has to be kept in mind that the rigidity of the tongue should be taken into account with these observations.

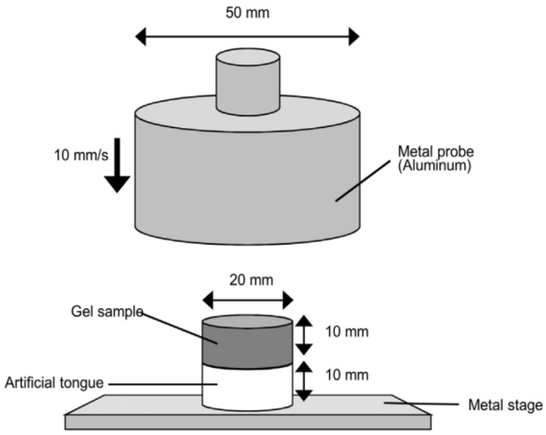



**Figure 2.15:** **Compression of agar gels over artificial tongues made of silicone (Ishihara et al., 2014).**

Kohyama et al. (2021) performed similar experiments on artificial tongues made with urethane and with varying stiffness. The authors observed that the stiffness of the tongue played a vital role in the fracture of hard and soft gellan gels (Figure 2.16). One can observe that the fracture points of either hard or soft gels were very distinctly different when compressed over a glass plate (GP) or over the softer tongue mimics such as so-called C0. This study hence demonstrates the feasibility of adapting classical texture analyzer with an artificial tongue to demonstrate the fracture phenomena of soft gels between the tongue and hard palate. The fracture properties obtained from these tests could hence be useful in designing food textures that can be manipulated by tongues of varying strengths.

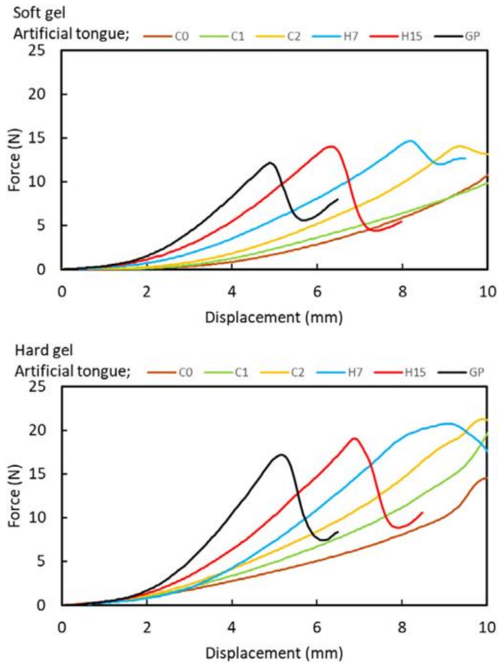
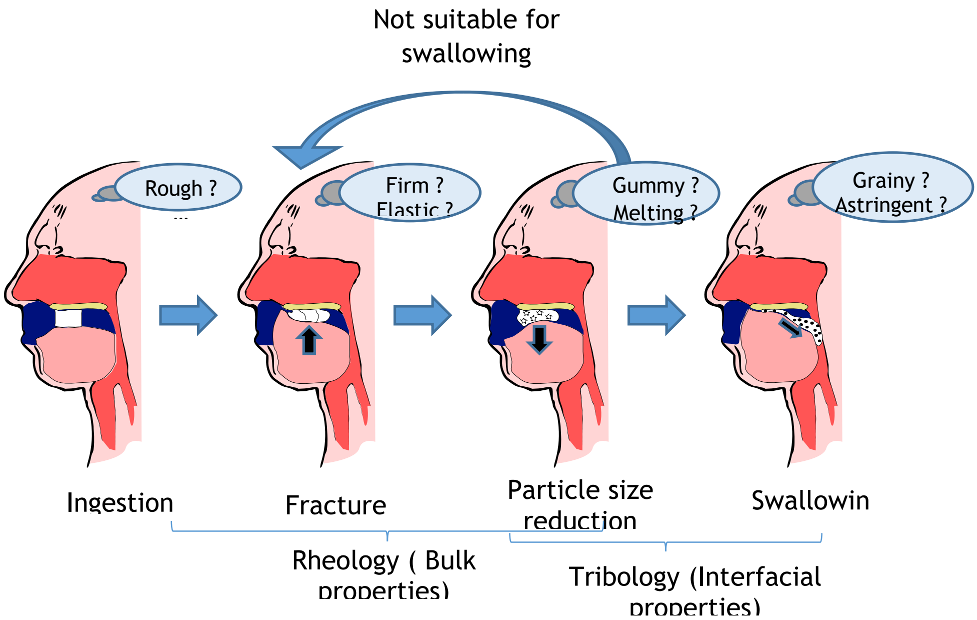



**Figure 2.16:** **Compression of gellan gels over artificial tongues made of urethane (Kohyama et al., 2021).**

As highlighted in the model proposed by Hutching and Lillford (1988), it has been known for a long time now that food undergoes significant transformations during the oral process (particle size reduction and hydration by saliva). These transformations have been partly described from a rheological point of view and successfully related to certain mechanisms of texture perception. However, we can see the increasing need to take into account the mechanical properties of the different parts of the oral cavity involved in the mechanical destructuring. Moreover, Stokes et al. (2013) also showed the importance of mechanical interactions between the food and the different components of the oral cavity, with a growing interest in tribological approaches particularly relevant at the end of the oral process where lubrication theory can provide new insights (Figure 2.17).




**<u>Figure 2.17:</u> Evolution of mechanical phenomenon during oral processing (Stokes et al., 2013)**

## <u>2.3.2.2 Tribology</u>

Tribology is the domain of physics which looks into the phenomena of friction, lubrication and wear between surfaces in relative motion. In regards to the food oral processing, the tongue interacts with other oral surfaces when shearing the food, and hence the role of surface properties becomes evident (Chen and Stokes, 2012; Stokes et al., 2013). Unlike conventional tribology, the surfaces involved in oral processing are soft hence it would be more appropriate to name it as soft tribology. It has emerged as one of the main techniques for understanding the friction phenomena between soft oral surfaces and food during oral processing (Krop, Hetherington, Holmes, Miquel, & Sarkar, 2019; Liu, Stieger, van der Linden, & van de Velde, 2015; Stokes, Boehm, & Baier, 2013).

The friction coefficient (μ) is the parameter measured in tribology, and it is expressed as the ratio of tangential force and normal force. The friction coefficient evolves with the normal force and the shearing velocity applied between the surfaces, and with the rheological properties of the fluid at the interface (in particular with viscosity). Moreover, in the case of oral tribology, the tongue and the palate are also deformable and rough, which has an impact on the friction coefficient.

Pradal & Stokes (2016) have underlined that tribology depends on the whole systemand not just on the lubricant (food/saliva) or on the surface alone. According the authors, the variation of friction coefficient depends on (i) the contact geometry of the system (ball-on-disk or pin-on-disk with sliding or rolling motion), (ii) the surface properties (hard or soft, rough or smooth, hydrophilic or hydrophobic), and (iii) the lubricant (food/saliva) properties (rheology, matrix heterogeneity etc).

Frictional data are commonly analyzed by Stribeck curve where friction coefficient is plotted as a function of the entrainment speed. This curve although was first built for hard surfaces but also holds true for soft contacts. It describes three regimes of lubrication namely, boundary, mixed and hydrodynamic regimes. The regimes of lubrication, as seen in the figure 2.18, are dependent on the lubricant film thickness. Many reviews have discussed Stribeck curve and its dependence on different parameters (Ahmed, 2018; Pondicherry, Rummel, & Laeuger, 2018). However it should be noted that especially for food samples, there could be deviations from standard Stribeck curve. The reason behind such observation lies in the fact that due to the deformability of the tongue, its topology may change according to the pressure fields to which it is subjected. Furthermore, pressure fields can also cause irreversible changes in the complex and heterogeneous microstructure of the food, affecting its rheology.

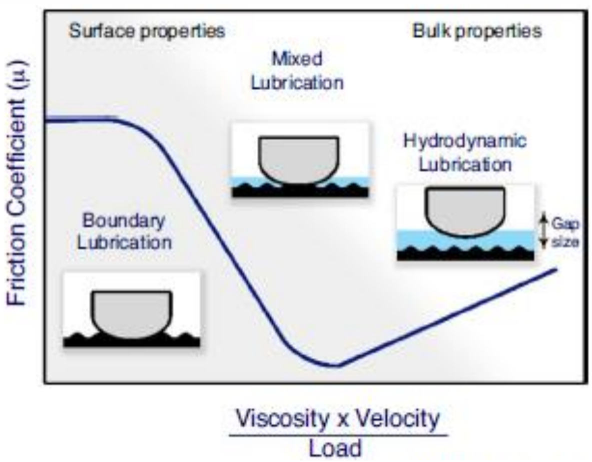
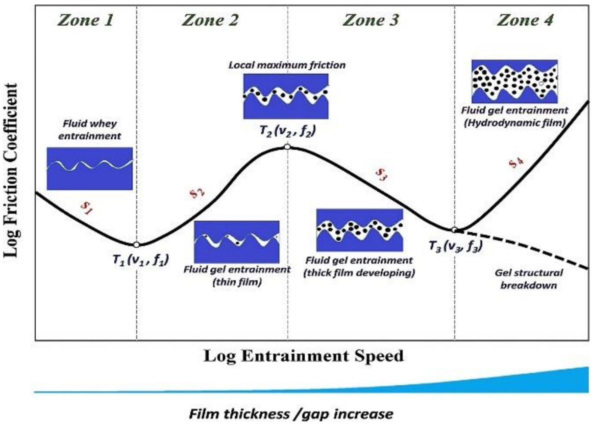



**Figure 2.18:** **Schematic representation of the Stribeck curve (Rudge et al., 2019).**

As an example, the study by Nguyen, Kravchuk, Bhandari, & Prakash (2017) presented the tribological characterization of yogurts. Results (Figure 2.19) were obtained using a ring-on-plate geometry on a hydrophobic surface. The obtained Stribeck curve can be divided into four zones (unlike classical ones). These zones are formed due the heterogeneous nature of the yogurt, consisting of fat as the dispersed phase. Observing the curve from left to right one can notice the transition from zone 1 to 4, in the first, only the fluid is between the two surfaces with fat droplets in the bulk. As the entrainment speed increases with increase in gap size, the fat droplets from the bulk enters the gap which increases the friction. When the speed was further increased, the gap size was considerably large to form hydrodynamic layer, and friction decreased. This observation clearly holds up the statement by Pradal & Stokes (2016) that tribology is system dependent.




**<u>Figure 2.19:</u> Stribeck curve for yogurt (Nguyen et al., 2017).**

<u>Adapting tribological measurements to mimic oral conditions</u>

The type of tribological surface is therefore just as important as the type of food in oral tribology. When looking for surfaces mimicking human tongue, one should keep in mind its physiological and anatomical complexity (as discussed in the previous section 2.1). The characteristics of the tongue like bulk rigidity and roughness might have a complex and synergistic role in oral tribology; they must therefore be well considered in experimental set-ups.

Researchers have tried to recreate real physiological parameters by using biological tongue tissues or artificial polymer surfaces that can mimic the wettability and deformability of the tongue (Carpenter et al., 2019; Dresselhuis, de Hoog, Cohen Stuart, & van Aken, 2008). However, there are some difficulties associated with these novel surfaces as well:

- Once biopsied, biological tissues rapidly lose their mechanical properties, making it difficult to preserve the tissue's surface profile and rigidity.

- Artificial surfaces, such as those made with polydimethylsiloxane (PDMS), need to be carefully modified to properly replicate surface roughness, rigidity, and wettability. PDMS has emerged as the most widely used material to accommodate the deformability of the tongue. The rigidity of PDMS surfaces can be altered, but often in number of tribological studies the rigidity of the surface was significantly higher than that of human tongue (Campbell, Foegeding, & van de Velde, 2017; Winkeljann, Bussmann, Bauer, & Lieleg, 2018). PDMS is inherently of hydrophobic nature, but the surface wettability can be modified using certain techniques like hydrophilization by oxygen plasma treatment (S. Lee & Spencer, 2005).

The surface roughness profile is of great importance in any tribology study. As any polymer, the PDMS can also be imparted with the desired roughness by molding it in a rough mold. However, the challenge is how to replicate the tongue surface profile as the data of surface characterization of human tongue is sparse.

Gaining grounds in this field, researchers (Wang, Wang, Upadhyay, & Chen, 2019; Figure 2.20) have developed techniques to replicate human tongue surface by creating its negative molds which are then used to imprint the same on the desired tribological surface. Wang et al. (2019) investigated tongue surface profile of 71 young participants. Double molding procedure was used (Figure 2.20) : first, a negative mold using silicone rubber was created directly by molding the tongue asperities, followed by imprinting it on gypsum for the replication of the surface microstructure of human tongue. The three dimensional data were generated by optical scanning technique. Authors observed that the tip of the tongue was the roughest part. The peak density although gradually decreased from front to back. The arithmetic roughness (which is the parameter of height of peak) at the tip of the tongue was reported between 50 µm to 160 µm.

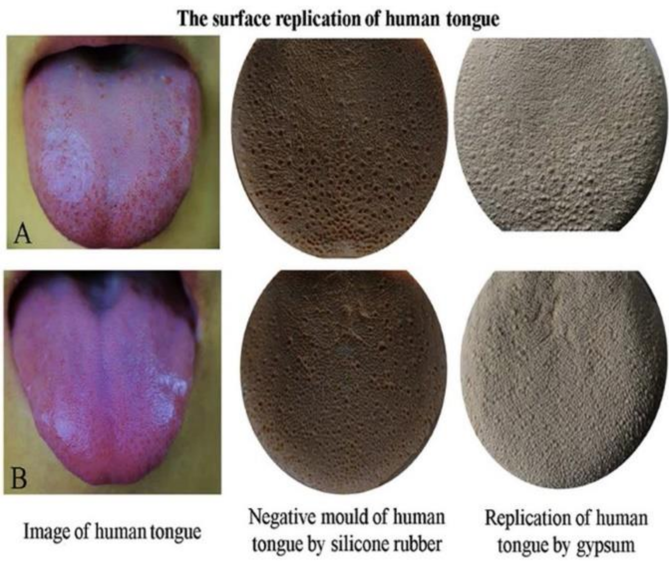
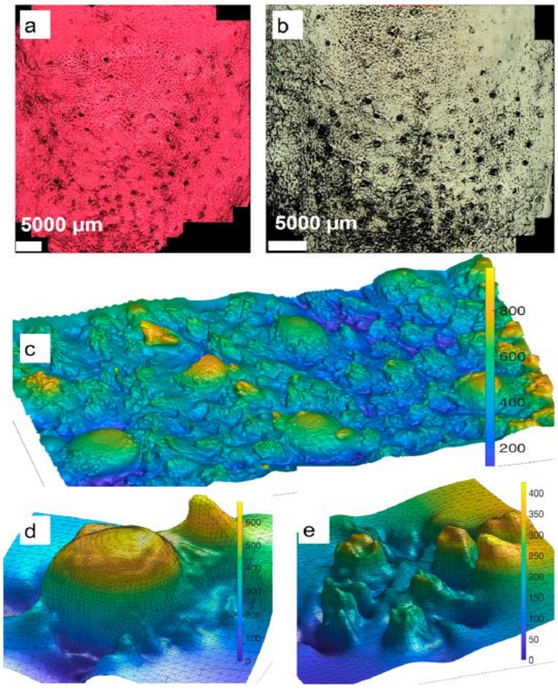



**Figure 2.20: Replication of tongue surface replication using a negative mold of silicone (Wang et al., 2019).**

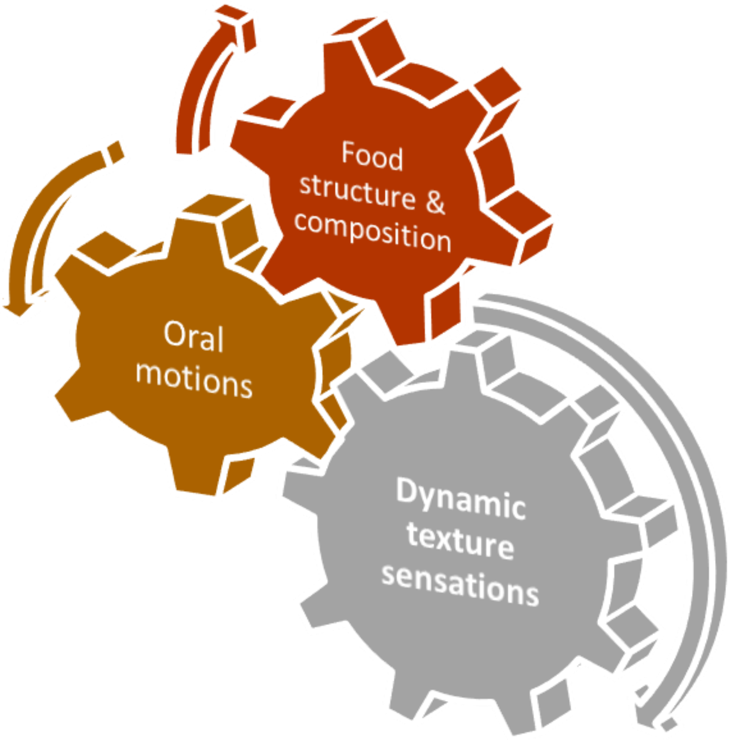
More recently for the first time, Andablo-Reyes et al. (2020) fabricated 3D printed molds replicating the tongue surface topology (Figure 2.21). Similar to the aforementioned study, where negative molds were generated, here negative optical scans were created. Then, with these scans, a random papillae distribution was modelled and 3D printed, With the these molds tongue mimicking samples made of soft silicone with modified wettability properties (both hydrophilic and hydrophobic) were created. The study also underlines the importance of considering random patterns of asperities rather than regular ones like grids. Hence we can say that these efforts to fabricate new biomimetic surfaces are paving the way for better quantification of the mechanical interactions between food and oral surfaces.




**<u>Figure 2.21:</u> Characterization of human tongue surfaces using polymeric impressions. Negative 3D optical scans of the tongue impressions taken using (a) hydrophobic (polyvinyl siloxane) and (b) hydrophilic (alginate) masking materials where the papillae are visible as circular holes. (c) Positive 3D optical image using polydimethylsiloxane (PDMS) mask of the human tongue obtained using the hydrophobic impression shown in panel a. (d) Soft-lithography technique showing zoomed image of fungiform papillae with their characteristic dome shape (e) filiform papillae as crown-shaped aggregate of pillars. The meshes of tesselated triangles in panels d and e are generated using Screened Poisson surface reconstruction of the point data sets obtained from the 3D optical scanning conforming accurately to the surfaces of 3D objects (Andablo-Reyes et al., 2020)**

Although it should be noted that with all the literature discussed in this section, the strategy of analyzing the tongue-food-palate system altogether and the associated interfaces is the key to unfold the complexities of food oral processing and texture perception. Saint-Eve et al. (2018) in this depiction (Figure 2.22) have also tried to summarize how the oral anatomical and physiological characteristics along with food structural and mechanical properties drive the perception of texture.

Accounting for the complexity of tongue rigidity or surface properties has been shown to be of interest for studying different stages of food oral processing, such as tongue-palate compression for breaking gels, of tongue-palate shearing when lubrication theory becomes relevant. However, there is a need for methods that make it possible to monitor the behavior of bulk and




**<u>Figure 2.22:</u> Dynamics of texture perception and its guiding principles.**

interface properties all along food oral processing, and not specifically during one particular stage. Moreover, if the method can be noninvasive and nondestructive, it can definitely be of great advantage, with the perspective, upon long-term, to use it in vivo, during real food consumption by individuals.

## <u>2.3.2.3 A novel ultrasound based technique</u>

Recently, quantitative ultrasound (QUS) based techniques have been proposed to quantify and visualize the evolution of tongue-food interface in real time during oral processing. Ultrasonic (US) waves are high frequency acoustic waves that make it possible to separate and analyze the mechanical response of the media and interfaces they propagate through. The advantage of low intensity ultrasound methods is that they are portable, non-invasive and non-destructive. As a consequence, they have a high potential for in vivo dynamic investigations. Ultrasound imaging techniques are widely used in the biomedical field, and have been proposed to study tongue movements during oral processing (Gao et al., 2013; Gao & Kohyama, 2014). They consist in positioning a multi-element ultrasonic sensor under the chin of the consumer, and in observing in real-time the echographic response of the tongue-food-palate system. Ultrasound examinations rely on the characterization of the energy backscattered by the different interfaces encountered by the beam in the tongue-food-palate system. Beyond the investigation of tongue motion during speech and oral processing, these recent studies reveal the feasibility of US waves propagation in such heterogeneous media. On the other hand, ultrasonic methods can be dedicated to the mechanical characterization of the media they propagate through. The analysis of wave propagation phenomena (reflection, transmission, refraction, scattering, absorption) with signal processing techniques make it possible to retrieve parameters of interest (like speed of sound, reflectivity, attenuation) that are representative of the mechanical properties of propagation media and interfaces. QUS techniques have thus been used for the real-time characterization of food products and of transformation processes (Awad et al., 2012; McClements, 1995). In the same manner, the compression of food between the tongue and palate can be assimilated to a process of food transformation. Efforts have thus been made to investigate how QUS techniques can be used for the real-time and in situ monitoring and characterization of this process. However, the heterogeneity of the tongue tissues, the geometrical irregularities of the oral cavity, oral tissues, temperature variations, or the complex nature of the tongue movements are all factors that strongly impact the phenomena of ultrasound wave propagation. Thus, the decision was made to develop the method in a controlled environment, using a biomimetic system (Figure 2.23, Mantelet, 2018). The advantage of this approach was that it allowed to integrate gradually different aspects of the complexity of the physiological environment, and to analyze their respective contributions to the ultrasound response.

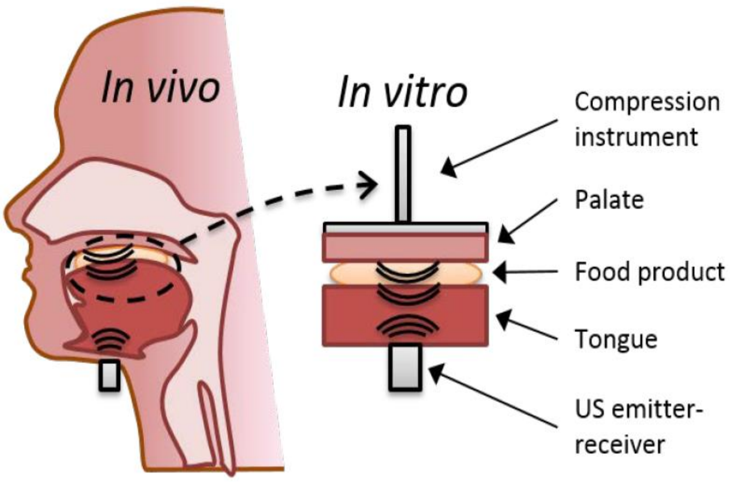



**Figure 2.23:** **Conceptualization of the biomimetic setup.**

Mimicking the impact of tongue roughness during a compression

The biomimetic device was then conceptualized to mimic the compression of the food between the tongue and palate in order to study the potential of the ultrasound method to decode the role played by some specific oral physiological characteristics on the mechanisms of texture perception (Mantelet et al., 2020a and b) In particular, attention was focused on studying the impact of two key properties of the tongue: surface roughness and lubrication.  The main element of the device was a traction-compression device (TA.XT plus, stable Micro System, Surrey, United Kingdom), used to impart uniaxial stress between the tongue and palate. Although the movements between the tongue and the palate during the oral process are more complex, this simplified configuration was chosen for the proof of concept of the ultrasonic method. In this system, a circular aluminum probe (diameter 40 mm) was used to mimic the hard palate.

**Figure 2.24: Uniaxial compression set up with PVC based tongue mimic.**

Polyvinyl chloride (PVC) cylinders were used as the tongue mimicking surfaces (TMSs). Given the stress levels associated with compression between the tongue and palate, PVC can be considered as non-deformable. Moreover, it is easy to machine into the desired shape. For a first attempt, the choice of a non-deformable material was made to only focus on surface properties and not on material deformation. The velocity of ultrasound waves in PVC (about 2400 $m.s^{-1}$) has the advantage of being low for a solid material, although it remains significantly greater than velocity values encountered in muscle tissues (about 1500 $m.s^{-1}$). Different surface roughness profiles were considered (Figure 2.25), which were imparted on the PVC cylinders by sandpaper. The surface profile was characterized using two classical parameters: the arithmetical mean height, Ra (mean height of the asperities), and the correlation length, β (width of the asperities).

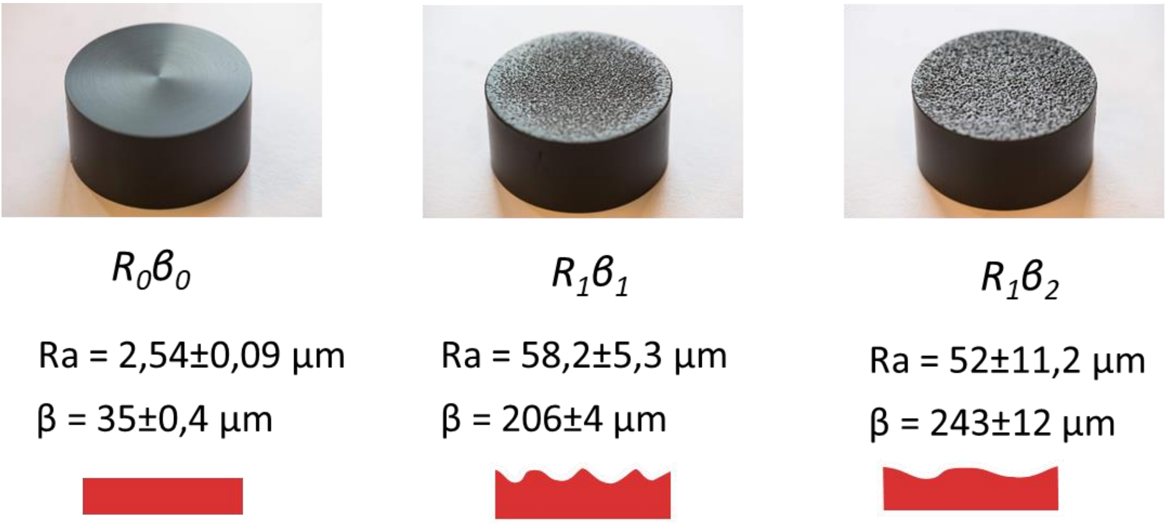



**Figure 2.25:** **Types of PVC based tongue mimic used by Mantelet et al. (2020a and b).**

Underneath the TMS, a 1 MHz transducer was placed, which emitted and received US waves (Figure 2.26). Model gels made from agar and gelatin were used the model food for the uniaxial compression. These model foods had varying composition of the polymers which resulted in very different Young's moduli.

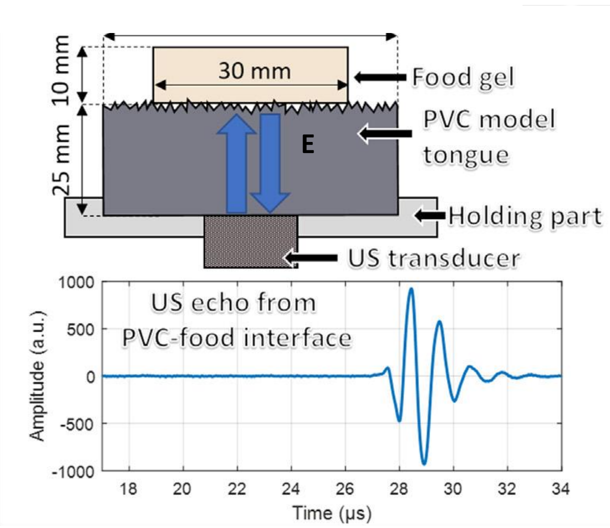



**Figure 2.26:** **Emission and reflection of ultrasound when encountering tongue-food interface.**

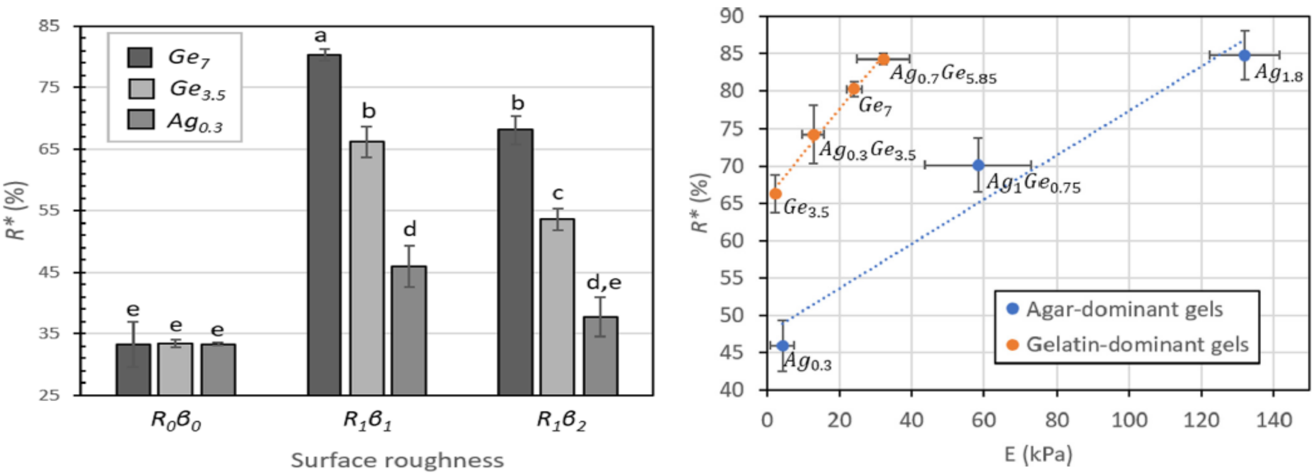
The apparent reflection coefficient (R*) of ultrasound waves at the interface between the TMS and the gels was introduced to assess the contact mechanism between the food and tongue. As seen in the figure (2.26), when US waves propagate in the system composed of a PVC based TMS on which a food gel is placed, a part of the acoustic energy is reflected back to the transducer after the wave meets the interface between the TMS and the food. The proportion of energy of the incident ultrasound wave reflected at this interface is obtained by the relation:

$$R^* = \left(\frac{\mathrm{A}}{\mathrm{A_{ref}}}\right) x\ 100$$

Where A and $A_{ref}$ correspond to the amplitude of the reflected echo (E) when the food is in contact with the TMS and when the tongue is in contact with air (reference measurements performed before the beginning of the experiments and where the entire ultrasound energy is reflected) respectively.

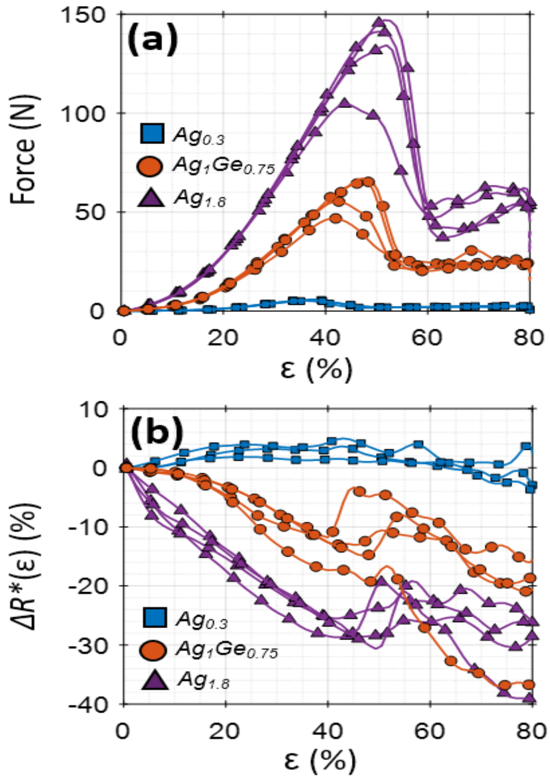
The reflection phenomena that cause these echoes are due to the differences in acoustic impedance of the media located on both sides of the interface. Acoustic impedance depends on: the mass density of the medium and the speed of the ultrasound waves in the medium. They can also be influenced by the intermediate presence of a third medium, even if it is very thin.

Firstly, this apparent reflection coefficient was studied in the case where the food is simply deposited on the TMS, without exerting any mechanical stress other than the gel's own weight. Some of the main results are discussed very briefly below:




**<u>Figure 2.27:</u> Mean values and standard deviations of the apparent reflection coefficient at the interface between TMSs and food gels.**

In the case of smooth TMS, it can be seen that the reflection coefficient is approximately the same, regardless of the type of gel. The value obtained, of the order of 35%, corresponds to the order of magnitude expected from the theoretical calculation which takes into account the differences in acoustic impedance between PVC and gels (the latter having very similar acoustic impedances). However, as soon as the surface of the TMS is roughened, drastic differences in R* are observed between the gels. This difference could be explained by the presence of air trapped at the interface between the TMS and the tongue, which is at the origin of a substantial increase in the reflection coefficient. Indeed, as air has a very low acoustic impedance compared to PVC, its presence leads to an increase in the acoustic energy reflected at the interface. The differences reported between the various gels made it possible to establish the impact of the rigidity of the gels (which reflects their capacity to follow the asperities of the TMS surface, and thus to limit the presence of air) and the type of polymer dominant in the gels (which conditions in particular their capacity to release a liquid film at the interface, and thus to promote the coupling between the TMS and the gel). The impact of a thicker or thinner water film at the surface (roughly mimicking the salivary film) has also been demonstrated.




**<u>Figure 2.28:</u> For the three agar dominant gels (*$Ag_{0.3}$*, *$Ag_1Ge_{0.75}$* and *$Ag_{1.8}$*): (a) variations of the force *F* applied during the compression, (b) variations of *ΔR** during the compression.**

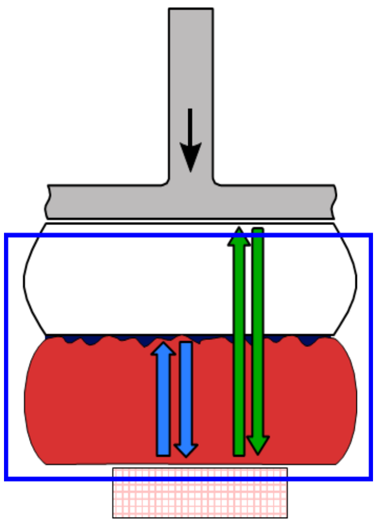
The main results thus confirmed the promising potential of the ultrasound method, making it possible to monitor in real time the reflectivity that reflects the evolution of the coupling at the interface between the TMS and the gels. In general, the trend observed was a decrease in R* as the compression progressed, as the stress fields exerted favored the deformation of the gels and their adaptation to the asperities of the tongue, but also as they contributed to the release and spreading of a liquid film at the interface. Interestingly, the work also reveals relaxation mechanisms at the interface, particularly as the gels fracture.

## 2.4. Objective of the thesis

In the backdrop of the review of literature performed, it can be highlighted that there is a need of developing instrumental methods which take into account the physiological and anatomical characteristics of the tongue for better comprehending the mechanical aspect of texture perception. Several novel approaches dealing with rheology and tribology have allowed considerable progress in the identification and understanding of different interactions between the tongue and the food, but only at specific stages of the oral processing. However, the need for the development of dynamic methods, in order to follow the tongue-food system in real time throughout the oral processing, and in a non-invasive and non-destructive way is still evident. In this context, a pioneering work has been conducted to introduce the use of quantitative ultrasound methods to characterize the physical phenomena occurring at the interface of the tongue and the palate on a bio-mimicking setup. **This work laid the foundation of my thesis work, which takes the next steps to build upon the proof-of-concept presented before and to reach towards more realistic oral processing scenarios.**

1. **Deformability of the tongue:**

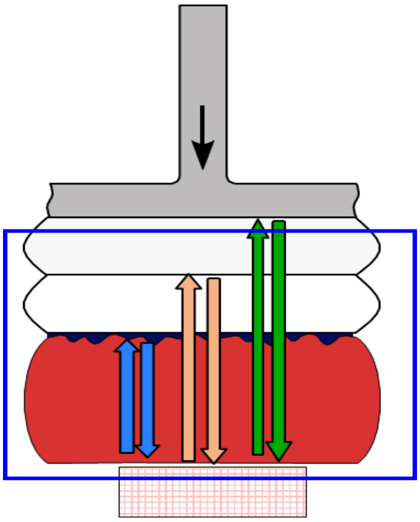
   To begin with, the first objective of my PhD work was to build an artificial tongue model that is more realistic to the real human tongue. The PVC tongue used in the previous work was non-deformable, and also its acoustical characteristics were not similar to that of human tissue. Hence, we made an informed choice of using a polymer called polyvinyl alcohol (PVA) for fabrication of the artificial tongue models. The choice of using PVA was based on advantages like scope of controlling rigidity, surface roughness and, hydrophilic nature. The PVA hydrogels have also widely used to mimic organs and help in the development of imaging techniques

   The deformability of the tongue in response to the compression of a food gels over its surface brings an extra dimension to the study, which is closer to reality. In this part, for the first time, we used the fluctuations of the Time of Flight of the US echo of tongue-food interface to monitor the deformation of the tongue. We were thus able to discuss it with the properties of the tongue like rigidity and surface roughness, but also with food mechanical properties. The detailed investigation on the deformability of the entire tongue-food system is discussed in Chapter 3 of this manuscript under the title: Ultrasound monitoring of a deformable tongue-food gel system during uniaxial compression–an in vitro study.

2. **Complexity of the food:**

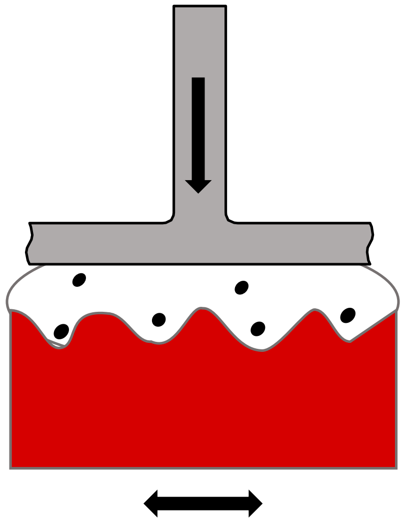
The second objective was to develop more complex food gels (macroscopically heterogeneous) and to investigate if the quantitative ultrasound method is still effective in heterogeneous day to day food. For this part of the thesis, different bilayer model gels were fabricated during my research exchange at Wageningen University. These bilayer gels were made of different stacking together monolayers made of agar and gelatin. Several combinations were studied with different polymers and fracture stress.



In this investigation, the ultrasound properties of the set-up were adjusted and it also required changes in signal processing methods for better tracing the reflection of echoes from heterogeneities inside the food. This new approach will allow us to understand how polymer type (agar or gelatin) affects the mechanical responses of bilayer gels uniaxially compressed over a biomimetic tongue. More specifically, the method could help to quantify the relative deformations of the individual components of the whole system: the artificial tongue model and the two gels layers. The detailed investigation is discussed in Chapter 4 of this manuscript under the title: Texture contrast: ultrasonic characterization of bilayer gel deformation during compression on a bio-mimicking tongue.

### 3. **Complexity of oral motions:**

The third objective was the key stepping stone to our effort in understanding the food oral processing. A new biomimetic setup was needed to be developed to incorporate shearing motions in addition to the uniaxial compression. With addition of shearing motion, friction phenomena occurring at the interface of tongue-palate were studied. The setup had to be designed to provide us control of parameters like shearing velocity, number of shearing cycles, normal force applied to the tongue-food system. To this was added of course the mastery of the mechanical and topological properties of the tongue, based on the expertise developed in the first two parts of the thesis with the PVA model. To complete this objective, a multi-disciplinary approach was required which included designing and assembly of structural element of the setup and programming a custom-made user-interface and related signal and data processing algorithms.



The feasibility of the set-up first was needed to be tested by measuring how friction coefficient values were affected by food properties, tongue properties, and operational parameters (normal stress, shearing velocity). The detailed explanation of the set-up and friction measurements during shearing of model food is discussed in Chapter 5 of this manuscript under the title: A new biomimetic set-up to understand the role of the kinematic, mechanical, and surface characteristics of the tongue in food oral tribological studies.

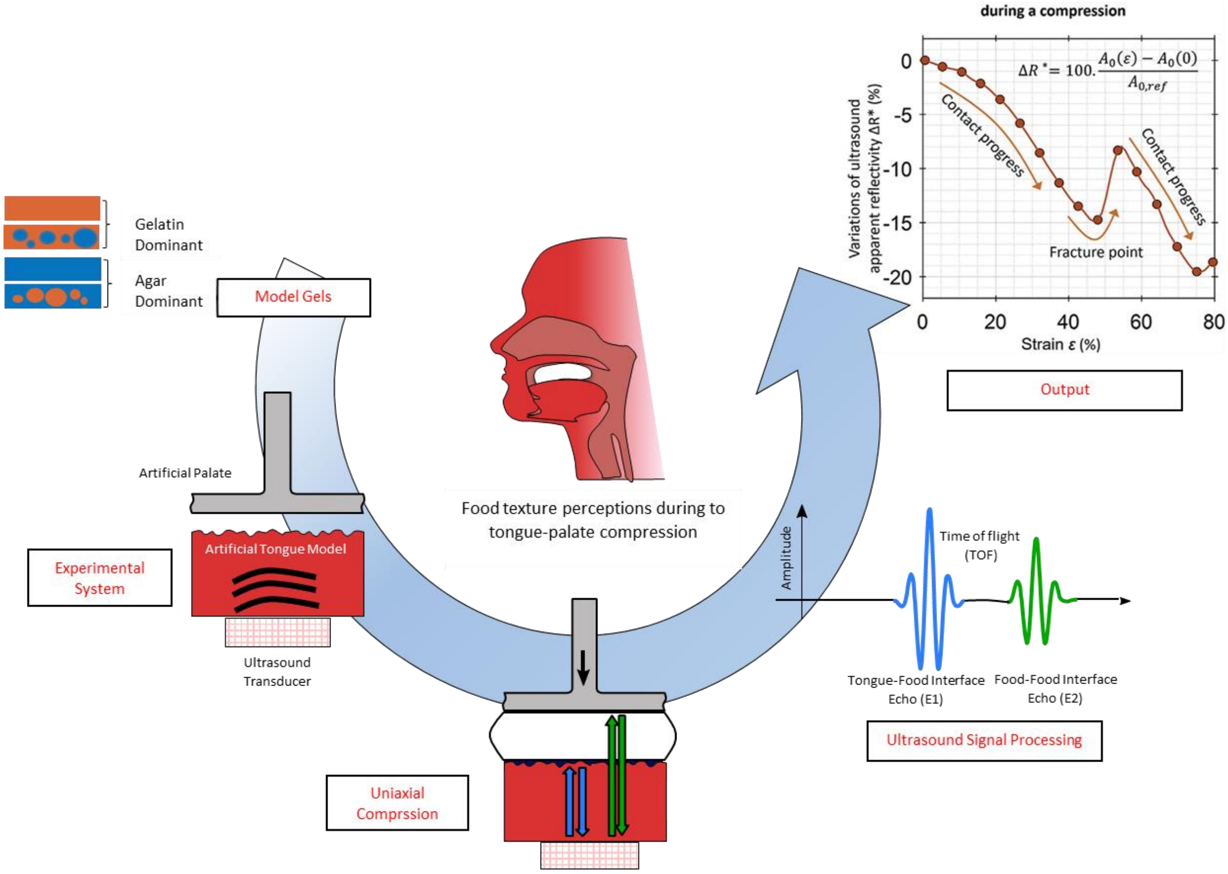
# III

# Ultrasound monitoring of a deformable tongue-food gel system during uniaxial compression–an in vitro study

# Preface

*As mentioned in the objectives, this thesis builds upon the experience recently gained from the proof-of-concept work for the use of quantitative ultrasound to track coupling mechanisms at the interface between a rigid and rough tongue mimicking surface and food gels. In this chapter, the ultrasound method was further extended for exploring the mechanical deformability of artificial tongue models (ATMs) topped with agar and/or gelatin food gels during uniaxial compressions. The ATMs here were made from polyvinyl alcohol and displayed different levels of rigidity and surface roughness. We collected real-time measurements via an ultrasound transducer placed beneath the ATM, enabling to monitor non-invasively the ultrasound time of flight (ToF) and the apparent reflection coefficient (R*) of the ATM-food interface. We were thus able to quantify deformations of the ATM-food system as well as coupling mechanisms at ATM-food interface, as it underwent compression induced by a texture analyzer. This chapter is presented in the form in which it was published in the peer-reviewed journal - Innovative Food Science and Emerging Technologies.*

# Graphical abstract



## 3.1. Introduction

Food texture perception is the result of complex and dynamic phenomena, which occur simultaneously during food oral processing. Understanding the dynamics of texture perception is hence of paramount interest for food scientists who seek to understand and model consumers' sensorial experience of food (Renard, Van De Velde, & Visschers, 2006; Wilkinson, Dijksterhuis, & Minekus, 2000). As technological advances are made, new and more precise techniques for characterizing food properties (e.g., structure, rheology, and tribology) have been developed (Stokes, Boehm, & Baier, 2013). However, it remains difficult to precisely associate mechanical measurements with sensory attribute ratings and to follow texture dynamics during oral processing. The human oral cavity is very complex and equipped with different types of mechanoreceptors, which can detect both static and dynamic stimuli. The tongue is a crucial organ within the oral cavity: it moves food back and forth, facilitating proper mastication and the safe swallowing of boli. It also plays a very important role in texture perception, especially when food is compressed between the tongue and the palate (Ishihara et al., 2013; Kohyama, Ishihara, Nakauma, & Funami, 2021). During oral processing, the deformation of food over the tongue surface creates clusters of different stimuli, which are perceived by the mechanoreceptors and then sent to the brain for further processing, including texture determination. There is a growing body of literature that underscores how understanding the tongue's physiological properties can shed light on changes in food structure during oral processing and the resulting diversity of texture perceptions (Engelen & Van Der Bilt, 2008; Ketel, de Wijk, de Graaf, & Stieger, 2020; Van Vliet & Primo-Martín, 2011). Therefore, it is essential to further explore the dynamic interactions taking place between food and oral surfaces.

To effectively monitor such interactions, it is necessary to use a non-destructive technique that can be applied in real time. Ultrasound-based approaches have shown great promise and have been successfully employed to visualize tongue movement and bolus swallowing (Gao et al., 2013; Peng, Miethke, Pong, & Lin, 2007). Nevertheless, ultrasound images only provide qualitative information. Therefore, there has been a need to develop ultrasound-based methods with quantitative metrics that can more precisely reveal the occurrence of events at the tongue-food interface. However, quantifying the propagation of ultrasound waves in the human oral cavity can be quite difficult. There is thus a need to develop an oral biomimetic set-up where the behavior of ultrasound waves can be studied in a controlled manner. Mantelet, Restagno, Souchon, & Mathieu (2020) and Mantelet, Srivastava, Restagno, Souchon, & Mathieu (2020) developed a novel ultrasound method for analyzing the tongue-food interface *in vitro*; their research performed a detailed examination of ultrasound reflectivity at the tongue-food interface, characterizing the effects of food properties, lubrication, and tongue surface roughness. This proof-of-concept work utilized rough but non-deformable tongue-mimicking surfaces made of polyvinyl chloride (PVC). It was found that the reflectivity of ultrasound waves (as estimated via the apparent reflection coefficient, R*) at the tongue mimic-food interface could be exploited to monitor changes in contact over time. This research also clarified the influence of food properties and lubrication on R* and, thus, on interface interactions.

However, the PVC tongue-mimicking surfaces did not very realistically recreate an actual human tongue, and there was thus further room for improvement. Therefore, in this present article we have introduced a novel artificial tongue-mimicking model (referred to as an ATM hereafter) made of polyvinyl alcohol (PVA). In recent years, PVA has been used in biomedical engineering to model arteries and oral mucosa (Chatelin et al., 2014; Fromageau et al., 2007; Gennisson et al., 2007; Jiang, Liu, & Feng, 2011; Mamada, Fridrici, Kosukegawa, Kapsa, & Ohta, 2011). Tongue mimics made with PVA were also used in our previous study dealing with oral tribology (Srivastava et al., 2021). PVA is a water-soluble synthetic polymer and can be used to form hydrogels. The rigidity of PVA hydrogels can be controlled by varying the number and kinetics of the freezing and thawing cycles (Fromageau et al., 2007). This ability to control hydrogel rigidity made PVA a good fit for our study system.

The deformability of the PVA based ATM meant that it could be used in compression experiments in which changes in tongue thickness over time were quantified using an ultrasound parameter called time of flight (ToF). The data gathered helped reveal the relationships among tongue dynamics, food properties, and system deformation. Using the PVA based ATM, we also studied variability in R* in response to ATM properties (i.e., surface roughness, Young's modulus values, and water-release capacity) and food gel properties (i.e., Young's modulus values and water-release capacity), as such factors influence the tongue-food interface and, in turn, affect interactions between food and the tongue.

## 3.2. Materials and methods

### 3.2.1. Preparation and characterization of food gels

Preparation of the food gels

Eight different model gels (Table 3.1) made of agar and/or gelatin were prepared as described in Mantelet et al. (2020a and b). First, a sucrose solution (white sugar [Cristalco, Paris, France] dissolved in water) was prepared via stirring at 20 °C for 30 min. If needed, agar powder (HP700IFG, Kalys, Bernin, France) was incorporated, and the mixture was heated to 100 °C (until agar was completely dissolved) in a flask sealed with aluminum foil to limit any loss of water. After the agar powder was completely dissolved, the mixture was cooled to 60 °C. This temperature was maintained, and gelatin (Bloom 250 PS 8/3, Rousselot, Gent, Belgium) was added (if required); the mixture was continuously stirred for 20 min. Once the gelatin was completely dissolved, the solution was poured into cylindrical molds (30 mm in diameter, 10 mm in height) made of polyethylene and exposed to conditions promoting gelation (20 °C for 15–18 h). The gel was unmolded just before the experiment to prevent any loss of water or shape over time. Table 3.1 summarizes the mechanical properties of the gels, which were characterized during the aforementioned research.

Water-release capacity of the food gels

The water-release properties of these food gels were also quantified using a protocol adapted from Sala, Stieger, & van de Velde (2010). Three Whatman™ filter paper disks (110 mm diameter; GE Healthcare Life Sciences, Chicago, USA) were placed on the base plate of a texture analyzer (TA.XT plus, Stable Micro Systems, Surrey, United Kingdom). A given gel was deposited on the disks and subjected to uniaxial compression (20% strain rate, 10 mm/s); there was a 5 s holding period. The maximum strain rate was chosen to avoid gel breakdown. The amount of water released was determined by comparing filter disk mass before and after the compression test. This methodology was somewhat modified in the case of the $Ag_{0.3}$ gel because the latter was very fragile and released water even without being compressed. Therefore, this gel was left on the filter disks for 5 sec and was then removed, without the application of any external stress. The paper was then weighed as in the case of the other gels. The results are presented in Table 3.1. However, in all future comparisons, we assumed that $Ag_{0.3}$ had highest water-release capacity.

**Table 3.1: Description of the food gels (Diameter: 30 mm; Height: 10 mm): composition (they all contained 15% wt of sucrose), Young's modulus values, and water-release capacity. Standard deviations are provided**

| Gel type | Composition (% wt) | | | | Young's modulus (kPa) | Water released (g) |
|---|---|---|---|---|---|---|
| | Water | Agar | Gelatin | TWEEN 20 | | |
| $Ag_{0.3}$ | 84.7 | 0.3 | - | - | 4 ± 3 | 0.150 ± 0.020* (without compression) |
| $Ag_{1}Ge_{0.75}$ | 83.25 | 1 | 0.75 | - | 58 ± 15 | 0.257 ± 0.020 |
| $Ag_{1.8}$ | 83.2 | 1.8 | - | - | 132 ± 10 | 0.151 ± 0.012 |
| $Ge_{3.5}$ | 81.5 | - | 3.5 | - | 2 ± 0.1 | 0.028 ± 0.002 |
| $Ag_{0.3}Ge_{3.5}$ | 81.2 | 0.3 | 3.5 | - | 13 ± 3 | 0.029 ± 0.004 |
| $Ag_{0.7}Ge_{5.85}$ | 78.45 | 0.7 | 5.85 | - | 32 ± 7 | 0.015 ± 0.003 |
| $Ge_{7}$ | 78 | - | 7 | - | 24 ± 2 | 0.015 ± 0.002 |
| $Ge_{7}T$ | 76.5 | - | 7 | 1.5 | 17 ± 2 | 0.012 ± 0.001 |

### 3.2.2. Preparation and characterization of the artificial tongue models

Preparation of the artificial tongue models

First, PVA powder (Sigma Aldrich, Saint-Louis, USA) was dissolved in ultra-pure water (at 10% w/w) under stirring for 2 h at 80 °C. The solution was then cooled until it reached room temperature. The solution was subsequently poured into cylindrical molds (50 mm in diameter, 20 mm in height) that contained abrasive paper at the bottom. The choice of ATM thickness is based on values reported in literature which ranged from 10 mm to 40 mm (Nakamori et al., 2020; Beghini et al., 2017; Ishihara et al., 2013). To simulate two levels of surface roughness, two types of abrasive paper were used: (i) P80 sandpaper (grain size = 200 µm; Struers, Champigny-sur-Marne, France) and (ii) P40 sandpaper (grain size = 425 µm; Norton, Saint-

Gobain, France). Hereafter, they are referred to as $R_1$ and $R_2$, respectively. The molds were sealed and frozen at -20 °C for 10 h. They were then thawed for 14 h at 20 °C. The freezing and thawing cycles were adjusted to attain the desired rigidity (Fromageau et al., 2007). Two types of ATMs were prepared: a soft ATM ($C_2$), which underwent two cycles, and a hard ATM ($C_6$), which underwent six cycles. After the completion of the requisite number of cycles, the ATMs were unmolded and stored in reverse osmosis treated water at room temperature (20 °C) for several months.

Mechanical properties of the artificial tongue models

ATM rigidity was measured using uniaxial compression tests carried out with a texture analyzer (speed: 10 mm/s; strain rate: up to 20%; at least 3 replicates performed). This protocol was adapted from the work of Gao, Nakao, Ishihara, Funami, & Kohyama (2016). The resulting Young's modulus values are presented in Table 3.2.

Water-release capacity of the artificial tongue models

The ability of the ATMs to release water under mechanical compression was also quantified. For each ATM, any surface water was gently absorbed with Kimtech$^{TM}$ absorbent wipes (Kimberly-Clark, Irving, TX, USA) until the tissue came away dry. Subsequently, three layers of absorbing Whatman$^{TM}$ filter paper (110 mm diameter; GE Healthcare Life Sciences, Chicago, USA) were placed between the texture analyzer probe and the tongue's rough surface to fully collect the water released. We chose to use a strain rate of 5% and a holding time of 5 sec to avoid any damage to the tongue's surface from the aluminum probe. The difference in paper mass was then determined as previously described to estimate water-release capacity (Table 3.2).

Surface roughness profiles of the artificial tongue models

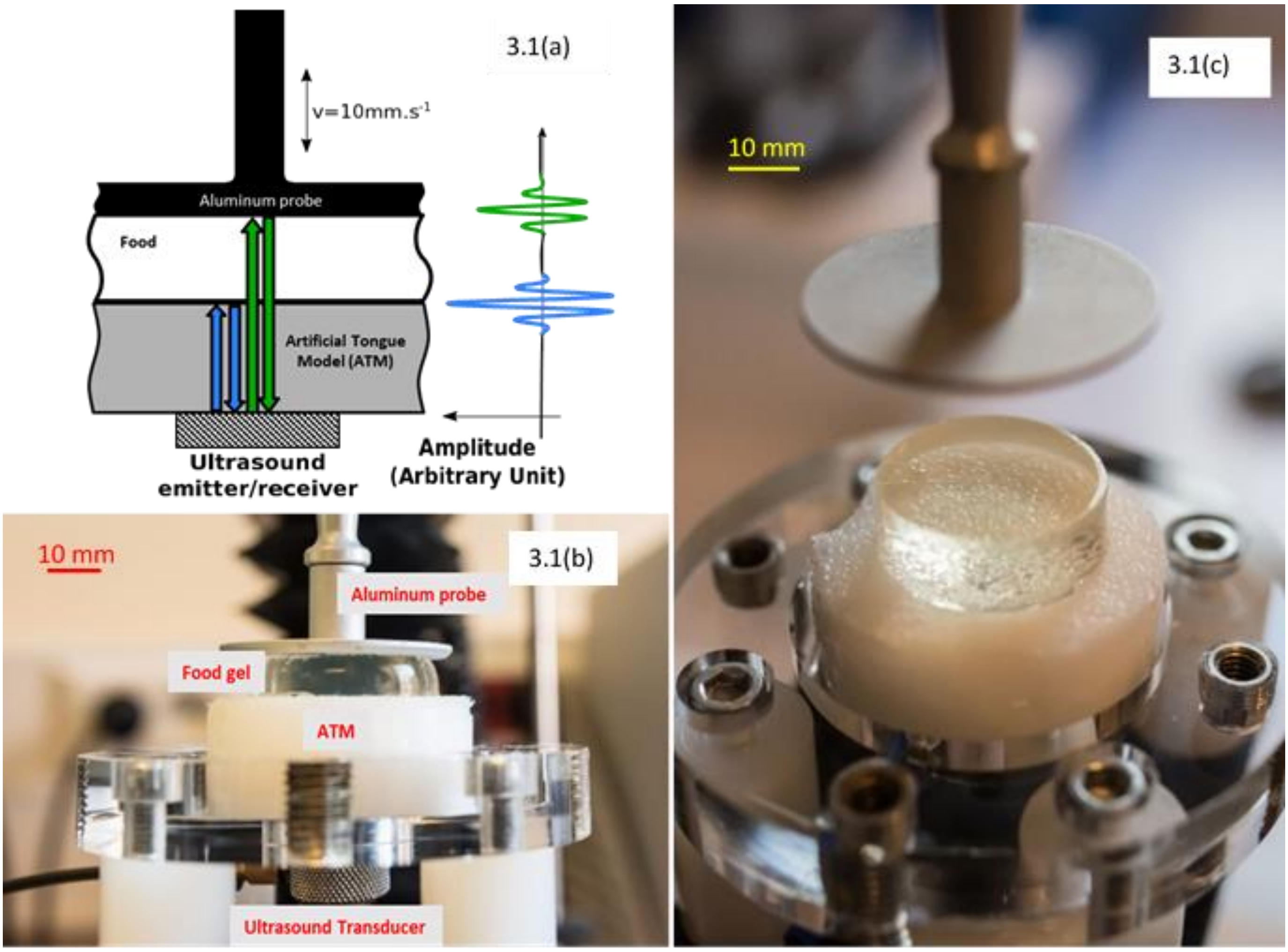
ATM surface roughness was analyzed via profilometry. Measurements were obtained using a contact profilometer (Dektak XT, Brucker, Billerica, MA, USA) with a 50-nm-radius stylus and 1 mg of applied force. For each ATM, three 30 mm surface scans were performed (one-dimensional, 45° of rotation between each scan). Vision 64 software (Bruker, Billerica, MA, USA) was used to control, analyze, and perform slope corrections before employing an algorithm in MATLAB (The MathWorks, Natick, Massachussetts, USA) to calculate roughness. Two parameters were chosen: the mean height of surface asperities (Ra) and correlation length, which is inversely related to asperity density (β). Although only two types of sandpaper were used, the freezing and thawing cycles had an impact on surface roughness as well. The values are presented in Table 3.2.

**Table 3.2: Description of the ATMs (Diameter: 50 mm; Height: 20 mm): Young's modulus values, surface roughness profiles, and water-release capacity. Standard deviations are provided when applicable.**

| ATM type | No. of freezing/ thawing cycles | Young's modulus (kPa) | Surface profile (µm) | | Water released (g) |
|---|---|---|---|---|---|
| | | | Peak height ($R_a$) | Correlation length (β) | |
| $C_2R_1$ | 2 | 23 ± 1 | 42 ± 1 | 289 ± 36 | 0.022 ± 0.002 |
| $C_2R_2$ | 2 | 18 ± 1 | 72 ± 14 | 435 ± 62 | 0.031 ± 0.003 |
| $C_6R_1$ | 6 | 71.2 ± 1 | 55 ± 3 | 341 ± 62 | 0.015 ± 0.002 |
| $C_6R_2$ | 6 | 71 ± 2 | 103 ± 3 | 429 ± 63 | 0.018 ± 0.003 |

### 3.2.3. Ultrasound measurements

As in Mantelet et al. (2020a and b), the ultrasound set-up (Figure 3.1) comprised a texture analyzer, a tongue-mimicking surface (a PVA based ATM in this study), and a mono-element piezoelectric ultrasound transducer (central frequency of 1 MHz; V103RM, Olympus, Shinjuku, Tokyo, Japan); the latter was placed beneath the ATM. Before starting each experimental test, the water on the surface of the ATM was gently removed using absorbent wipes (Kimtech™, Kimberly-Clark, Irving, TX, USA) until the tissue came away dry. The food gels were gently unmolded and placed on top of the ATM. The texture analyzer was used to apply a controlled uniaxial deformation (of up to 8 mm at 10 mm/s) to the ATM-food gel system using a circular aluminum probe (diameter: 40 mm). Ultrasound measurements were made prior to the food being placed on the ATM (i.e., reference measurements) and in real time during the compression period. To this end, the transducer, acting as both an emitter and a receiver (pulse-echo mode), was used in tandem with a pulser-receiver (Sonatronic, Evry, France). The pulser-receiver system was used to generate negative square wave pulses (width: 500 ns, amplitude: 80 V) and to digitize the radio frequency (*rf*) signals corresponding to the system's pulse echo response (12-bit quantification, 100 MHz sampling rate, 38 dB gain). The frequency of pulse recurrence was around 90 Hz. A LabVIEW (National Instrument, Austin, Texas, USA) interface was used for real-time acquisition of the *rf* signals during the compression period.




**<u>Figure 3.1</u>: (a) Schematic representation of the experimental set-up; Pictures of (b) the polyvinyl alcohol artificial tongue model (ATM) and (c) the ATM topped with a food gel.**

### 3.2.4. Signal processing

Similarly to the work done in our previous research Mantelet et al. (2020a and b), the apparent reflection coefficient of ATM-food interface (R*) was calculated on all the ultrasound signals of each test. ATM deformability had an impact on both the amplitude and the time of occurrence of the ultrasound waves reflecting off the ATM-food gel interface. The deformations induced during compression shortened the path to be covered by the waves. As a consequence, the time needed for the waves to travel between the emission source and back was shorter as well. Thus, in addition to R*, here we have used another ultrasound parameter: time of flight (ToF).

Each experimental test is composed of a set of signals which have been analyzed using Matlab. Figure 3.2a shows the typical shape of an *rf* signal, composed of two main echoes: $E_0$ for the tongue-food interface, $E_1$ for the food-palate interface. Figure 3.2b shows the M-Mode representation of the whole set of signals acquired during a test, where signal amplitude is coded in color and thus allows to trace the evolution of the echoes identified in Figure 3.2a. For each signal, noise reduction was implemented via a low-pass filter (cut-off frequency: 10 MHz). To precisely characterize ATM deformation, it was crucial to accurately determine the ToF for the echo, $E_0$, associated with the ATM-food gel interface over the entire course of a given test. $E_1$ echo was not processed for this study. Using the reference signal, a time window was automatically defined; it had a width of 5 µs and was centered with respect to the Hilbert-transformed peak amplitude of the signal. This window of reference was then cross-correlated with all the *rf* signals acquired during a given test, allowing the automatic detection of the ToF associated with $E_0$.

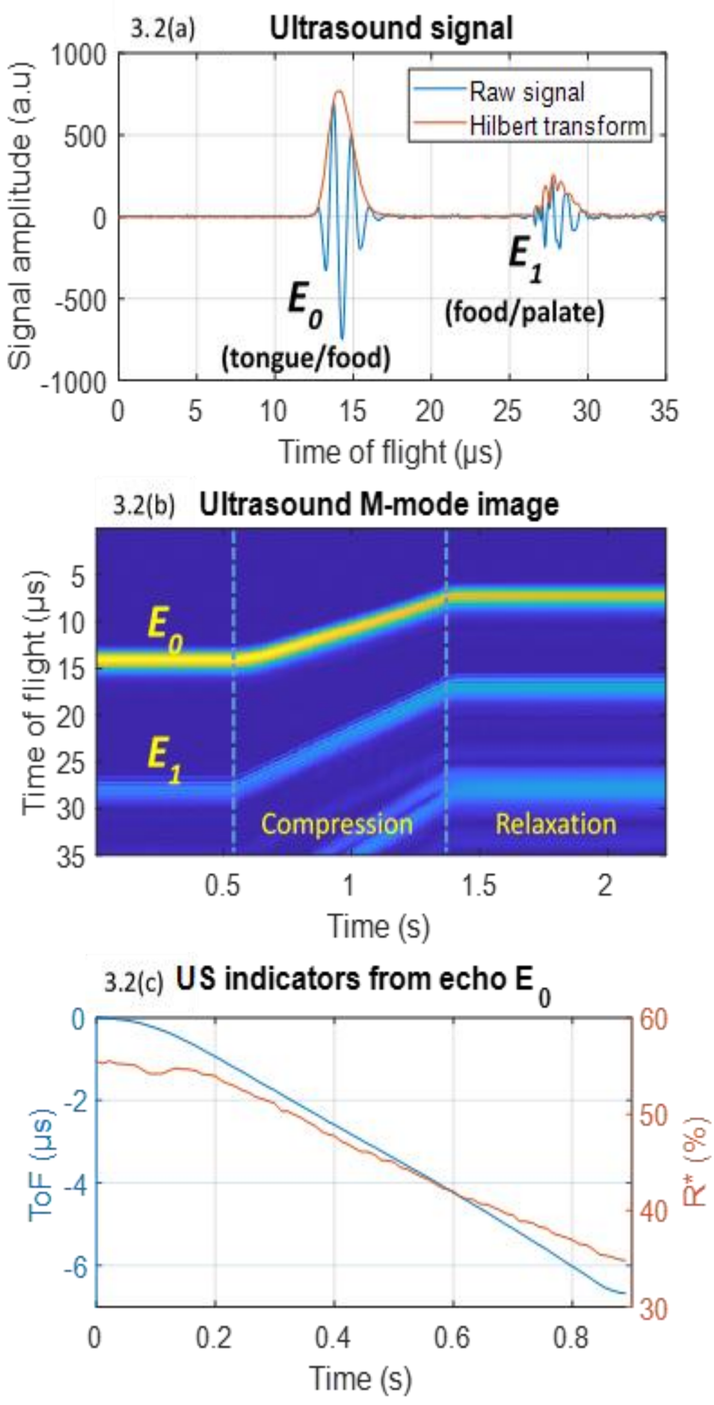



**Figure 3.2: (a) A typical *rf* signal, composed of two main echoes: $E_0$ for the tongue-food interface, $E_1$ for the food-palate interface; (b) Ultrasound M-mode imaging of the entire set of signals obtained during an experiment and (c) Variation of ToF and R* (calculated on the basis of $E_0$) during a compression.**

The ToF associated with the echo $E_0$ corresponds to the time required for the ultrasonic wave to return to the sensor after being reflected at the interface between the ATM and the food. The thickness (e) of the ATM is proportional to this time of flight ToF:

$$e = \frac{1}{2}.ToF.c$$

Where c is the propagation speed of ultrasound waves in the 10% PVA hydrogels (1540 m/s at 20 °C; Gennisson et al., 2007). Finally, the deformation of the ATM (Δe) was deduced by subtracting the real time thickness (e) of the ATM from its initial value $e_0$. As an example for validation of the accuracy of the method, we compared it with deformation measurements assessed with the texture analyzer. To do so, we considered the compression of an ATM without any food. The deformation applied was 20%, with a velocity equal to 10 mm/s (similarly to as done for the tests in presence of food). Three repetitions were performed. The obtained average curves with standard deviation envelopes can be compared in Figure 3.3. The obtained graphs hence show that the deformation measurements by both ultrasound and texture analyzer are in agreement, confirming the relevance of the ultrasound velocity value (1540 m/s) used for the calculation, and validating the method for tongue deformation assessment.

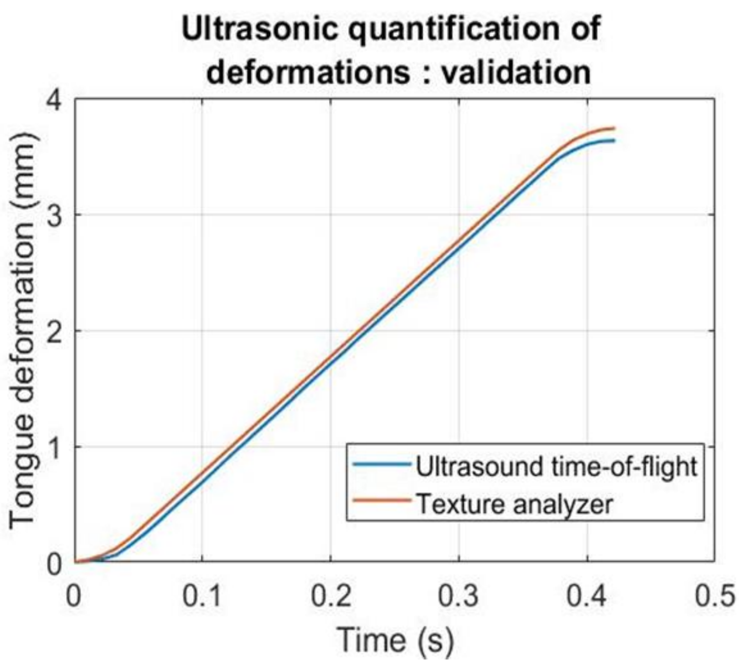



**Figure 3.3: Estimation of the tongue deformation (without food) by ultrasound time of flight and texture analyzer.**

To reliably calculate R* at the ATM-food gel interface, it was necessary to accurately estimate the amplitude of $E_0$. For all the *rf* signals, time windows encompassing $E_0$ had a width of 5 µs and were defined so as to be centered around the ToF for $E_0$. A Hanning window was applied, and the amplitude of $E_0$ was defined as the maximum amplitude of the power spectrum (the Fourier transform of the autocorrelation function) for the window obtained. R* was defined as $E_0$'s amplitude during a given test, expressed as a percentage of $E_0$'s amplitude during the reference measurements (i.e., when there was no food gel on the ATM). Consequently, the evolution of ToF and R* were computed for the entire duration of an experimental test, as illustrated in Figure 3.2c which focuses on the compression step.

## 3.3. Results and discussion

To begin with, the biomimetic set-up with different ATMs was developed. The roughness and rigidity values (Table 3.2) of the ATMs fabricated for this study were found to be in accordance with the values reported for real human tongues. Ishihara et al., (2013) has reported the rigidity of the human tongue at rest as 12.2 ± 4.2 kPa and at contracted state as 122.5 ± 58.5 kPa. Moreover, the surface asperities height (filiform papillae) of human tongue was reported between 50 - 150 µm (Andablo-Reyes et al., 2020; Wang, Wang, Upadhyay, & Chen, 2019). Also, Andablo-Reyes et al. (2020) reported average diameter of filiform papillae around 350 µm. Hence the characteristics of the developed ATMs were in good accordance with the values published in literature on human tongues.

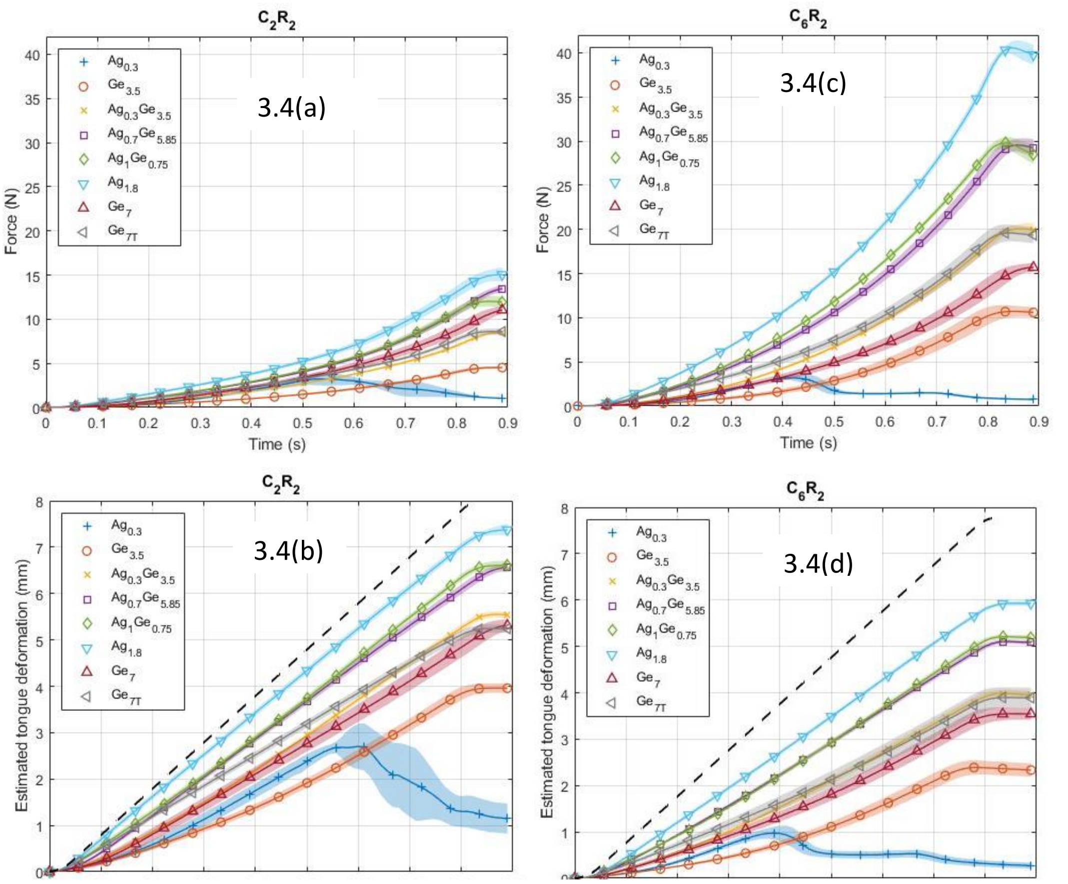
The results and discussion on these ATMs are discussed below and have been structured into two main parts that each focus on one of ultrasound parameters: (i) ToF and (ii) R*.

### 3.3.1. Characterizing the deformation of the artificial tongue models—ToF

It is important to investigate the deformation of the tongue during oral processing because this phenomenon can directly change food bolus characteristics and stimulate the mechanoreceptors responsible for texture perception. In this study, our *in vitro* approach allowed us to measure the ToF of the ultrasound waves reflected off of the ATM-food gel interface, providing an accurate estimate of ATM deformation during the uniaxial compression of the system. The maximum possible degree of ATM deformation (in the absence of food) was 40% because initial ATM thickness was 20 mm, and the level of deformation applied to the system was fixed at 8 mm. It is also important to have in mind that the diameter of the ultrasound beam (less than 10 mm) is lower than the diameter of the gels. Thus, the deformations measured by ultrasound only reflect what is happening in the central part of the ATM, which can be assumed as planar.

<u>Effect of food properties</u>

When the soft ($C_2R_2$) ATM was used, varied profiles of force (as measured by the texture analyzer) were observed over the range of food gels (plate displacement: 8 mm at 10 mm/s; Figure 3.4a). The ToF values obtained during these experimental tests showed that ATM deformation changed over time (Figure 3.4b). Force and ATM deformation showed similar trends. When tests were performed using the hard ($C_6R_2$) ATM, we saw the same patterns and similarity in metric values (Figures 3.4c and 3.4d). These results are encouraging because they underscore the relationship between ATM deformation and the force applied to the system during compression. The *in situ* and non-invasive characterization made possible here with ultrasound gives promising long term *in vivo* perspectives. Forces and deformations indeed constitute very relevant information to monitor during oral processing, but they remain impossible to characterize so far. The ToF values also showed that it was possible to identify the fracture point of food gels during compression. Like the level of force, tongue deformation was found to be sensitive to the sudden breakage of food gels. Out of the eight food gels, $Ag_{0.3}$

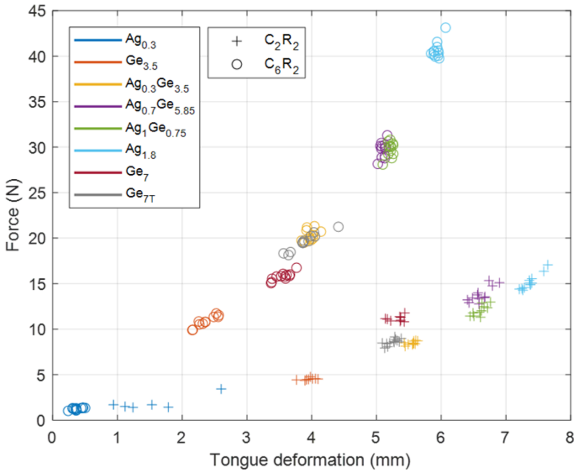



**<u>Figure 3.4:</u> (a) Force and (b) estimated ATM deformation over time as food gels were compressed on the soft ATM ($C_2R_2$); (c) Force and (d) estimated ATM deformation over time as food gels were compressed on the hard ATM ($C_6R_2$). The cloud around each line represents the standard deviation; at least six replicates were performed for each experimental test. The markers of different shapes were used to enhance the readability of the graphs. The dotted line on ATM deformation plot represents the maximum deformation imposed by the texture analyzer.**

was the only one that fractured during compression, and this event was easily observed in both the ATM deformation results and the force results. It is also clear from the results that ATM deformation was affected by the mechanical properties of the food gels. For example, because $Ag_{0.3}$ and $Ge_{3.5}$ were softer, they did not cause as much ATM deformation as did the more rigid food gels like $Ag_{1.8}$. To understand this phenomenon, it is useful to make the comparison with the situation of two springs connected in series. Total deformation is the sum of the strains experienced by the individual springs. Hence, the softer gels more equally shared the deformation resulting from system compression, whereas the more rigid gels did not, leading the ATM to deform more in compensation.

<u>Effect of artificial tongue model properties</u>

The human tongue is a complex muscular organ that is also a hydrostat. In other words, its volume remains constant when its rigidity is modulated or when it is in motion (Napadow, Chen, Wedeen, & Gilbert, 1999). Tongue rigidity changes considerably over the course of oral processing to better manage the bolus and texture perception. We used the PVA based ATM to investigate the role of rigidity in food deformation. We measured force and ATM deformation during experimental tests in which the different food gels were placed on the soft and hard ATMs ($C_2R_2$ and $C_6R_2$, respectively; values following compression: Figure 3.5).




**<u>Figure 3.5:</u> Force and estimated ATM deformation following compression (time $t_1$) for the experimental tests conducted with all the food gel types on ATMs differing in rigidity ($C_2R_2$ = soft and $C_6R_2$ = hard). Each color corresponds to a certain food gel type, whereas the two symbols distinguish between the ATM types.**

ATM rigidity affected the relationship between the level of force and ATM deformation. For both ATM types, the different food gels formed clusters that were based on the gels' mechanical properties). It represents that the diversity in mechanical property of gels were suitable for this study. Some food gels, like $Ag_{0.3}$, resulted in little tongue deformation, while others, like $Ag_{1.8}$, led to nearly 8 mm in deformation in the soft ATM, suggesting that the food gels themselves were not deformed at all. Taken together, the clusters of points for each ATM type followed a specific pattern, which was comparable to the curves obtained for the Young's modulus values during ATM compression. It is thus evident that the hard ATM led to greater force than did the soft ATM. Moreover, ATM rigidity also influenced food deformation kinetics. Using $Ag_{0.3}$ as an example, both the force and ATM deformation results show that gel fracture occurred earlier on the hard ATM than on the soft ATM (Figure 3.4). These results highlight that ATM rigidity plays an important part in shaping the extent and kinetics of food deformation.

The human tongue has a particular surface topology due to the presence of different papillae. However, the latter's role in the bulk deformation of food has yet to be thoroughly explored. In this study, force and ATM deformation displayed different patterns over time when certain food gels (notably gelatin-dominant gels) were placed on ATMs with the same rigidity but different surface roughness. For example, in case of the gelatin-dominant gels $Ge_{3.5}$ and $Ge_7$, the effect of surface roughness on tongue deformation was much more pronounced for the hard ATM (Figures 3.6a, 3.6b), since the deformation curve for $C_6R_1$ was more distant from $C_6R_2$ when comparing $C_2R_1$ with $C_2R_2$. The explanation could be that, when the cylindrical gels were compressed on top of the ATM, a barreling effect arose from friction and adhesion coming into play (Brennan & Bourne, 1994; Pons & Fiszman, 1996). Barreling effects occur when there is friction between a plate and a specimen, which results in a state of triaxial stress instead of the ideal state of uniaxial stress. Since gelatin polymers have adhesive properties and release less water, the degree of friction might have been greater. The agar-dominant gels did not display such a pattern, perhaps because of their higher degree of water release.

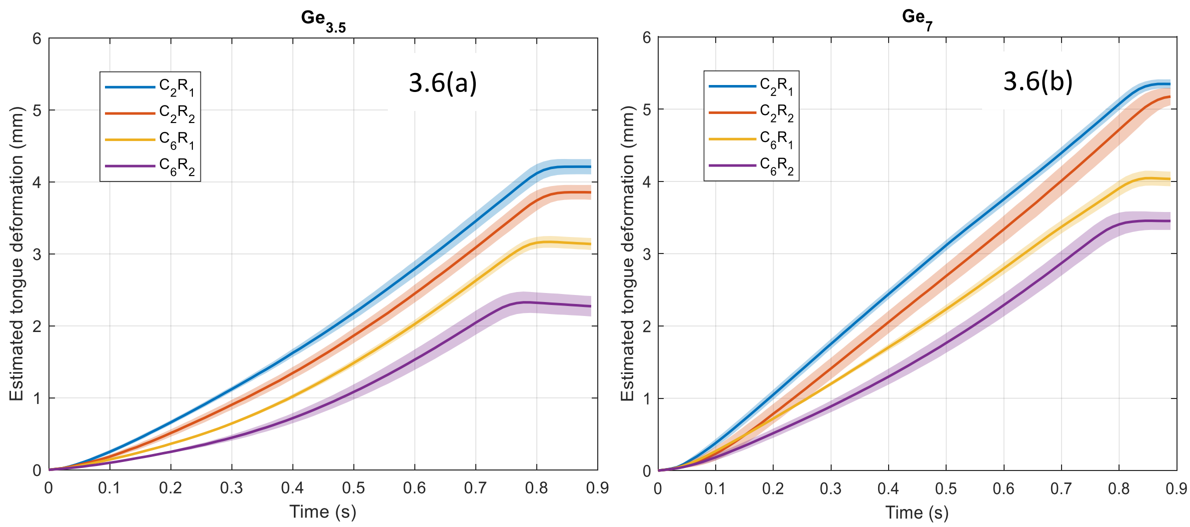
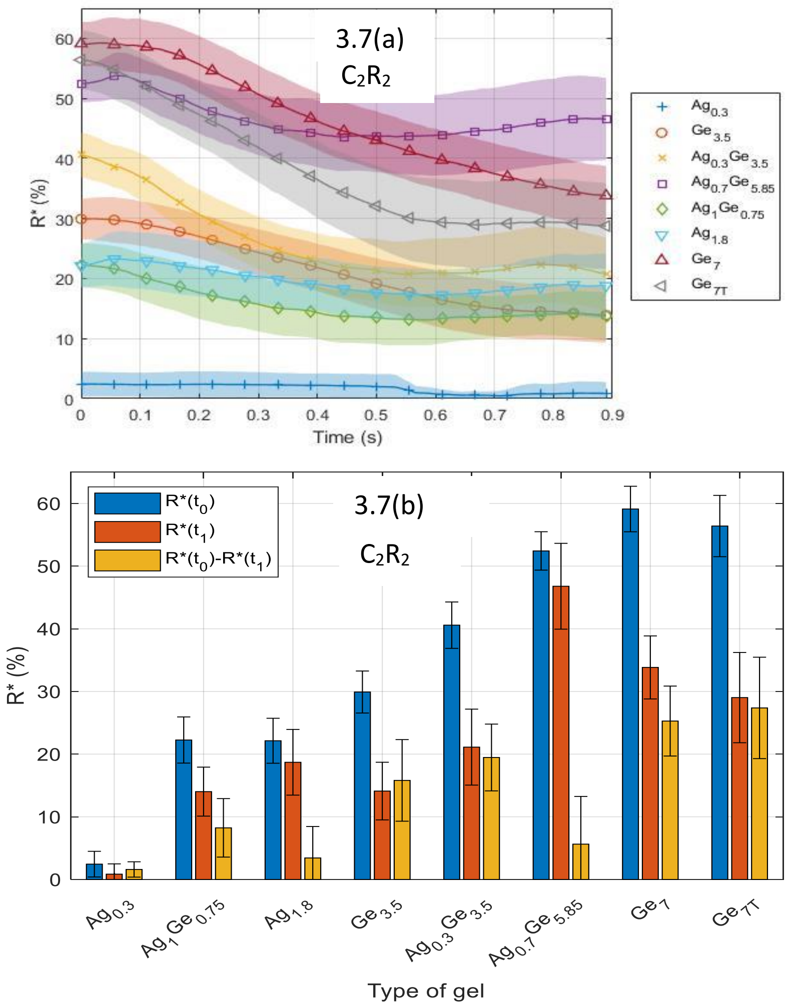



**Figure 3.6: Effect of surface roughness on estimated ATM deformation during the compression of the food gels (a) $Ge_{3.5}$ and (b) $Ge_7$. The cloud around each line represents the standard deviation.**

This explanation could also apply to the results for the hard ATM, which released less water during compression. Furthermore, the number of freezing and thawing cycles positively affected mean peak asperity height (Table 3.2). The presence of taller asperities with negligible water in the interphase region should have enhanced adhesion between the food gels and the ATM. Greater adhesion can enhance barreling effects, lowering the uniaxial force exerted on the system.

### 3.3.2. Analysis of the tongue model-food gel interface—R*

R* is the parameter used to quantify the apparent reflection of ultrasound waves at the interface between the ATM and the food gels, and its value is greatly dependent on the media's difference in acoustic impedance (Mantelet et al., 2019). R* has been shown to be a key parameter for understanding the characteristics of the tongue-food interface (Mantelet et al., 2020a and b). More specifically, it was found that there was a pronounced difference in acoustic impedance between a PVC tongue-mimicking surface and various food gels, resulting in the strong reflection of ultrasound waves at the interface. Here, in contrast, the PVA based ATM and the




**<u>Figure 3.7:</u> (a) R* values during the compression of the food gels on the soft ATM ($C_2R_2$); the cloud around each line represents the standard deviation; the markers of different shapes were used to enhance the readability of the graphs. (b) R* values before compression began ($t_0$), and after compression ended ($t_1$), and the difference between the two ($t_0$-$t_1$). The error bars represent the standard deviations.**

food gels appeared to have similar acoustical impedance, PVA being thus more representative of the acoustic properties of human soft tissue. However, it may also mean that the quantity of acoustic energy reflected by the ATM-food gel interface was limited. That said, there were clear differences in R* among food gels (Figure 3.7a). Results for the $C_2R_2$ ATM underscore the sensitivity of our ultrasound method when it comes to analyzing the interface between the PVA ATM (with its more biologically realistic properties) and the food gels. Previous research has examined variation in R* for a PVC tongue-mimicking surface (Mantelet et al., 2020a and b). The presence of trapped air at the interface was found to be a key factor affecting R* variations. The value of this parameter was at a minimum when no air was present, representing a state of perfect coupling between the ATM and food gel; in contrast, when R* was maximum, coupling was imperfect due to significant amounts of trapped air. Factors that can alter air presence are water release or mechanical deformation. The results obtained here were similar to those seen in the previous study. R* values varied depending on food gel type and the amount of water present at the interface.

Effect of food gel properties

Polymers like agar or gelatin form very different structures upon gelation, resulting in products that differ in rigidity and water-release capacity (Santagiuliana, Piqueras-Fiszman, van der Linden, Stieger, & Scholten, 2018). Consequently, we observed a variety of changes in R* over time: it decreased, plateaued, or increased over the course of compression, indicating the presence of a nonlinear response. It was therefore necessary to study the data quantitatively. When the values of R* were examined and compared for two time points (Figure 3.7b), $t_0$ (test begins—food gel is deposited on the ATM) and $t_1$ (test ends following the compression period), it was evident that two main factors were at play. Syneresis and the gel's ability to mold itself to ATM surface asperities appeared to govern changes in R* over time.

At $t_0$ (Figure 3.7b), the R* value for $Ag_{0.3}$ was quite low since the latter was an extremely soft food gel that released a large amount of water, improving contact with the ATM's surface. $Ge_{3.5}$ had a comparatively higher R* value despite its nearly equivalent Young's modulus value because it released less water. The values at $t_0$ can shed some light on why some food gels might be perceived as dry or wet when deposited on the tongue.

When the food gels were compressed on the ATM, the main trend observed was a decrease of R* since the application of stress tended to improve contact at the interface. The R* values following compression ($t_1$) provided much needed insight into the role of food gel and ATM properties in predicting the degree of improvement in contact. The higher the gel's gelatin concentration, the higher the initial R* value, suggesting poorer levels of contact. There was thus greater room for improvement when the uniaxial stress was applied. This difference was starker for the more rigid gelatin-dominant gels since the force exerted to achieve 8 mm in deformation was much higher, resulting in better contact between the food gel and the ATM.

Effect of artificial tongue model properties

ATM properties also influenced the R* values. When examining how ATM rigidity type ($C_2R_2$ = soft and $C_6R_2$ = hard) affected the results obtained with the food gels $Ag_{0.3}$, $Ge_{3.5}$, $Ge_7$, and $Ge_{7T}$, it was found that R* was higher at the start of the tests ($t_0$) for $C_6R_2$ compared to $C_2R_2$ (Figures 3.8a and 3.8b). The greater the number of freezing-thawing cycles, the denser and more rigid the PVA becomes, affecting the polymer's ability to release water. The resulting absence of water at the interface could explain why R* was higher for the $C_6R_2$ ATM. As discussed previously, low levels of lubrication at the interface can augment adhesion, resulting in a barreling effect and an uneven decline in compression force. This barreling effect might lead to uneven surface deformation as well as air pockets, thus increasing R*. Another explanation could be that the hard ATM underwent six cycles of freezing and thawing, more greatly affecting its surface roughness (peak height) even though sandpaper grain size was the same. Increased asperity height could have led to more air being trapped at the interface, boosting R* values.

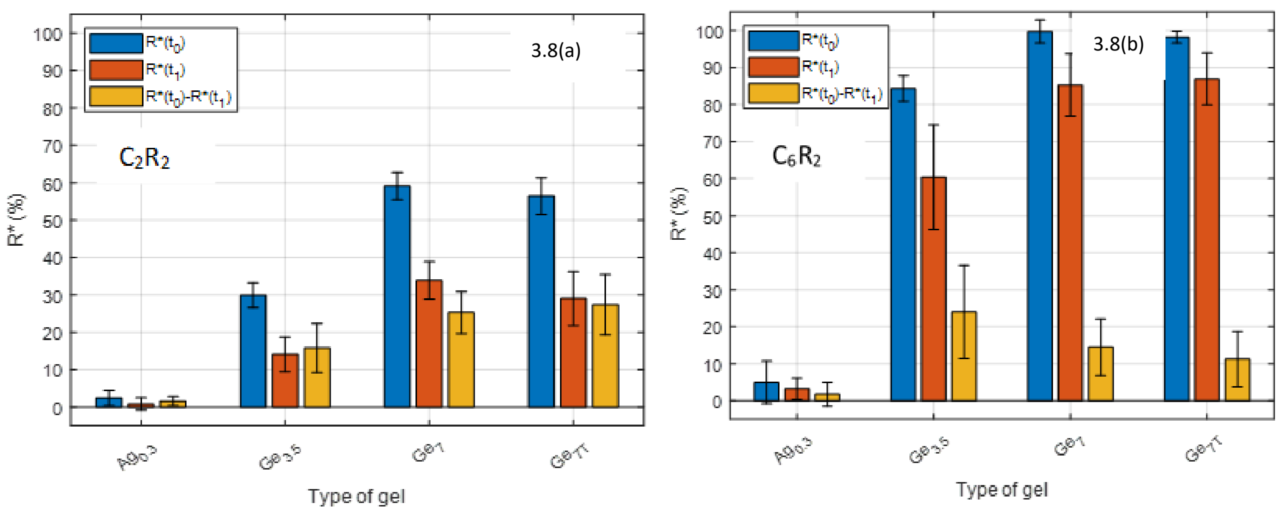
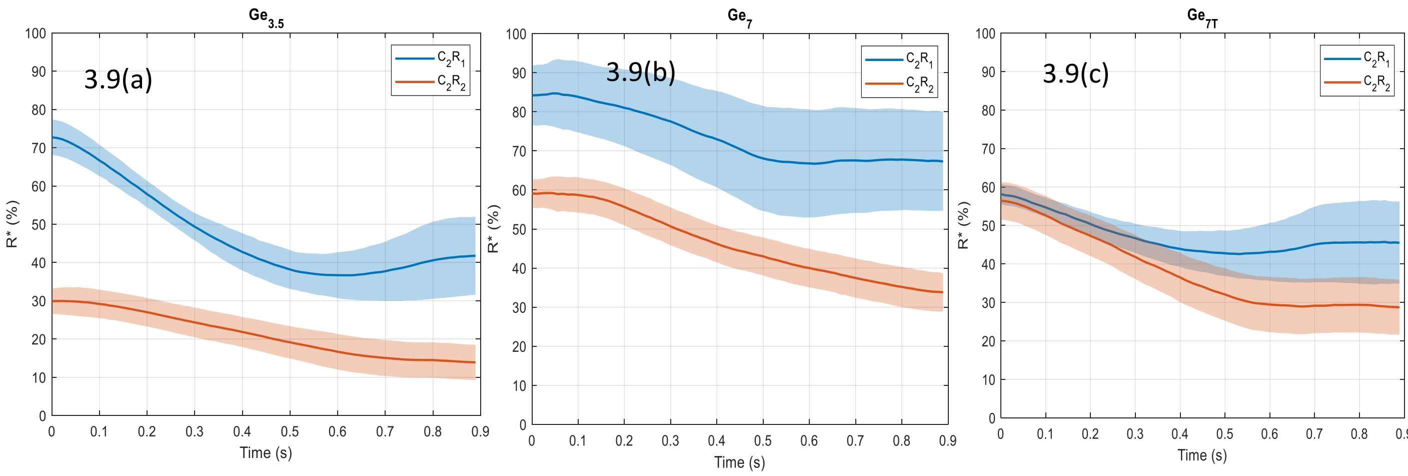



**Figure 3.8:** **Effect of ATM rigidity on R* during the compression of the food gels on (a) the soft ATM ($C_2R_2$) and (b) the hard ATM ($C_6R_2$). The error bars represent the standard deviations.**

When rigidity was controlled, ATM surface roughness ($C_2R_1$ vs. $C_2R_2$) was found to affect R* values (Figures 3.9a and 3.9b): it resulted in different temporal patterns in R* for the three gelatin-dominant gels ($Ge_{3.5}$, $Ge_7$, $Ge_{7T}$). R* values were higher for $C_2R_1$ than for $C_2R_2$, which was an unexpected result because one would assume that higher asperities would lead to more trapped air, enhancing wave reflection. However, it should also be noted that, compared to $C_2R_1$, $C_2R_2$ had a larger peak correlation length (β) (i.e., wider peaks) and hence a lower peak density. Peak density could affect surface topography overall, potentially explaining this unexpected observation.

The comparison of the R* values for $Ge_7$ versus $Ge_{7T}$ sheds light on the ability of TWEEN to modify interface characteristics (Figure 3.9c). As a surfactant, TWEEN lowers the surface tension of water, promoting its spread under conditions of compression and expelling the air trapped among asperities. It seems likely that the properties of the surfactant in $Ge_{7T}$ reduced the difference in the R* values when this food gel was used on ATMs of different surface roughness.




**<u>Figure 3.9:</u> Effect of surface roughness on R* during the compression of the food gels (a) $Ge_{3.5}$, (b) $Ge_7$, and (c) $Ge_{7T}$. The cloud around each line represents the standard deviation.**

## 3.4. Conclusions

In this study, we used PVA based ATMs with different bulk rigidities and surface roughness profiles to investigate the role of tongue deformability in the mechanical compression of food gels by a hard palate. The ToF of the ultrasound wave traveling to and from the ATM-food gel interface was used as a key metric for characterizing the overall deformation of the system. The ToF values helped estimate ATM deformation during the uniaxial compression of the food gels and were found to fit with the force exerted on the system. R*, or the apparent reflection coefficient, has been employed in previous studies to analyze tongue-food dynamics. It was found to be effective here as well for characterizing real-time changes at the ATM-food gel interface over the course of compression, including changes in the relationship between contact dynamics and both gel properties (i.e., Young's modulus values, water-release capacity) and ATM properties (i.e., deformability and surface roughness). More importantly, this study demonstrated that, despite the similarities in the acoustical impedance of the ATMs and the food gels, it was possible to accurately quantify reflection at a rather small scale. Consequently, this approach may hold promise for *in vivo* applications involving biological tissues and food, which have similar acoustical properties.

The study also underscored the utility of PVA in ATM design, given that it is a material that can better replicate tongue rigidity and roughness, a need that still exists in food oral processing research. Additionally, our findings showed that ultrasound techniques could be useful for tracking quantitative information related to mechanical interactions between the tongue and the palate.

Future work using this set-up will focus on investigating how ultrasound indicators respond when investigating systems with more complex, heterogeneous model foods. The broader goal is to progressively bridge the gap between model food and real food. Future research will also better account for the complexity of food oral processing, a greater diversity of motion (mixing shearing and compression) will be simulated and tongue shape, oral cavity anatomy and oral temperature will be more realistically recreated to further put ultrasound techniques to the test, prior to their more widespread use *in vivo*.

## Acknowledgements

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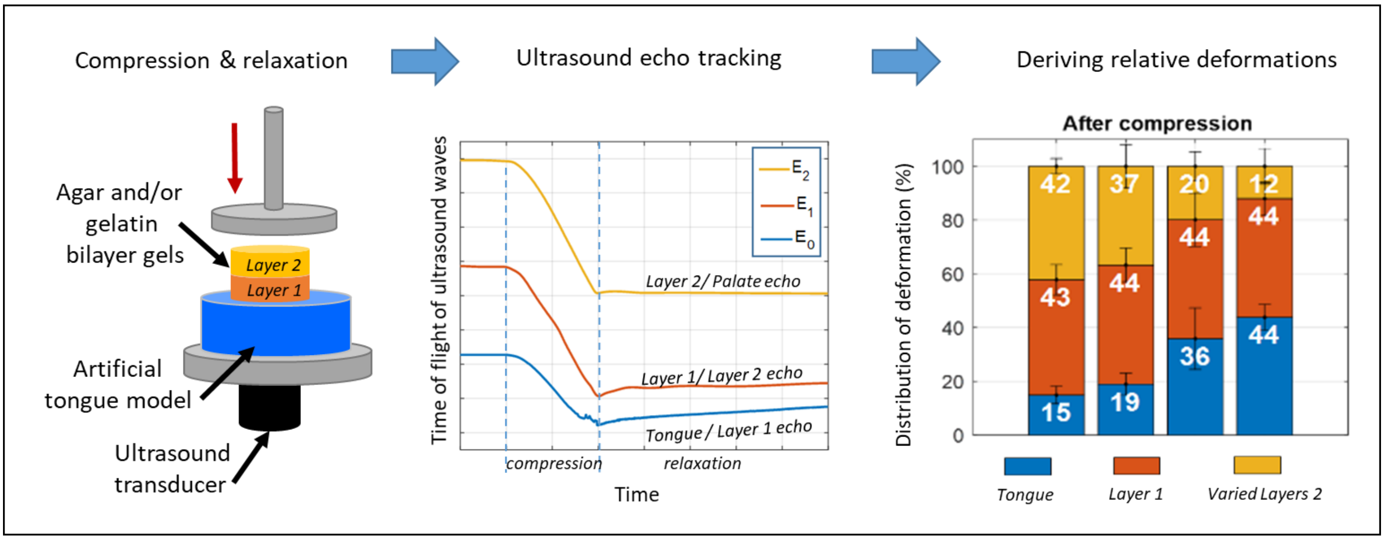
# IV
# Texture contrast: ultrasonic characterization of stacked gels' deformation during compression on a biomimicking tongue

# Preface

*In the previous chapter, the introduction of a PVA artificial tongue model allowed us to show that ultrasonic methods could not only follow the coupling mechanisms at the interfaces, but also the deformations endured by the tongue during uniaxial compressions. However, the foods we consume on a daily basis are much more complex than the homogeneous gels studied so far. The idea of the second objective of the thesis is thus to push a little further the study of the potential of ultrasonic methods by studying how they can inform on the mechanical behavior not only of the tongue, but also of composite foods.*

*For this purpose, bilayer gels were created using layers of brittle agar and elastic gelatin in different combinations. In a series of experimental tests, a bilayer gel was placed on a rough, deformable artificial tongue model (ATM) made of polyvinyl alcohol; a texture analyzer was used to apply uniaxial force, and deformation was monitored by an US transducer located under the ATM. This approach helped us to understand and quantify the synergistic deformation of each individual entity and their possible role in the perception texture contrast. This chapter is presented in the form in which it was recently published in the peer-reviewed journal - Current Research in Food Science.*

# Graphical abstract



## 4.1. Introduction

The solid and liquid foods that make up our diets have diverse structures and mechanical properties. Some have rather homogeneous structures. In recent years, homogeneous foods have been prominently used to clarify how they are processed in the mouth and how texture perceptions arise as a result. Studies have been carried out on actual foods, including breads, cookies, cheeses, and custards, to characterize food mechanical properties during oral processing (De Wijk, Prinz, & Janssen, 2006; Le Bleis, Chaunier, Della Valle, Panouillé, & Réguerre, 2013; Saint-Eve, Panouillé, Capitaine, Déléris, & Souchon, 2015; Young, Cheong, Hedderley, Morgenstern, & James, 2013). However, such foods only represent a small portion of our diets. Indeed, most foods are more complex; they are heterogeneous composites, combining different phases with highly contrasting mechanical properties. Altering such properties when formulating foods impacts the sensory experience of the consumer (Palczak, Giboreau, Rogeaux, & Delarue, 2020; Szczesniak & Kahn, 1984). Customizing food properties can also serve as a tool for improving nutritional quality (e.g., reduction of sugar, salt, and fat content or changes in digestive and nutrient-release kinetics) (Mesurolle, Saint-Eve, Déléris, & Souchon, 2013; Salles et al., 2017; Singh, Ye, & Ferrua, 2015). In recent years, interest has grown in creating model foods that display different levels of structural heterogeneity as a mean for understanding the phenomena behind the perception of texture contrasts (Devezeaux de Lavergne et al., 2016; Laguna & Sarkar, 2016; Larsen, Tang, Ferguson, Morgenstern, & James, 2016; Santagiuliana, Piqueras-Fiszman, van der Linden, Stieger, & Scholten, 2018; Santagiuliana, Bhaskaran, Scholten, Piqueras-Fiszman, & Stieger, 2019). In homogeneous foods, macroscopic textural properties can be rather easily verified and assessed. In contrast, in composite foods, the presence of structural heterogeneity presents a challenge: it is harder to predict texture perceptions because different structures have different mechanical properties. The individual structural components of a composite food may interact and deform synergistically during oral processing, making it difficult to trace the drivers of texture perception dynamics.

Devezeaux de Lavergne et al. (2016) studied how mechanical contrasts in stacked gels (two layers of emulsion-filled agar/gelatin gels with mechanical contrast) affected oral processing and texture perception dynamics, as measured via a sensory panel. Their results showed that, overall texture perception dynamics of the combined layers was the average of that of two individual layers. However, these interesting findings were difficult to interpret from a mechanical perspective because the layers' mechanical characteristics were quantified separately using a texture analyzer with a cylindrical probe.

Going a step further, Santagiuliana et al. (2018) used stacked gels (two layers) to investigate how the mechanical and physicochemical properties of individual gel layers influenced perceived heterogeneity. Here, interestingly, the mechanical characteristics of the stacked gels were directly measured using a penetration test that employed a texture analyzer with a wedge probe. Consequently, it was observed that the fracture force of the top layer had an impact on that of the bottom layer. The same study also investigated how differences in fracture stress between two layers affected the perception of texture contrasts. One of the main conclusions was that, depending on polymer type, a minimum degree of difference (5 kPa for soft and brittle agar layers; 16 kPa for soft and elastic gelatin layers) in fracture stress between the two layers was required for heterogeneity to be perceived. Taken together, these discoveries greatly clarified the bulk deformation dynamics that occur between different layers and the synergistic effects on the deformation kinetics of the entire system. It is thus evident that the distribution of stress/strain in each layer must be characterized during compression. To identify the layers' individual contributions, it is not enough to assess the system's overall mechanical properties using uniaxial compression tests; a complimentary approach is needed, in which real oral conditions, such as soft contacts between the palate and tongue, are mimicked.

Kohyama, Ishihara, Nakauma, & Funami (2019) presented an innovative approach of compressing soft food gels between an artificial tongue (transparent urethane gels) and plate, and monitored the deformation incurred by the gels with the help of video cameras. This approach was found very effective in analyzing the deformation and fracture phenomena in monolayer gels. However, they can pose some technical difficulties when several interfaces are stacked over each other. Depending on the color contrast between the media, or the level differences between the camera axis and the different interfaces, there may be some errors in the estimation of the deformations.

To overcome these limitations, ultrasonic methods can be a good candidate. Ultrasound based techniques being non-invasive and non-destructive, they have gained interest by the community of food science for the real-time and quantitative monitoring of various physical phenomena occurring during food processing (de Wijk, Wulfert, & Prinz, 2006). Indeed, ultrasonic waves are mechanical waves, sensitive to the mechanical properties of the media and interfaces in which they propagate. In addition to the well-known imaging approaches that allow to explore qualitatively the heterogeneities in the media, they can be used quantitatively to characterize properties (propagation speed, attenuation, acoustic impedance) revealing the mechanical characteristics of the propagation media. Two recent studies (Mantelet, Restagno, Souchon, & Mathieu, 2020a; Mantelet, Srivastava, Restagno, Souchon, & Mathieu, 2020b) described a method in which real-time quantitative ultrasound was used to explore coupling phenomena between food gels (agar and gelatin) and tongue-mimicking surfaces. Moreover in our recent study (Srivastava et al., 2021), a quantitative ultrasound method has been proposed to quantify the deformation dynamics of artificial tongue models during the compression of monolayer gels. The characterization of the tongue deformation was achieved by the use of single element transducers. Unlike multi-element imaging probes, the measurement of deformations with mono-element transducers does not face limitations induced by image resolution. Thanks to signal processing methods, they allowed an accurate estimation of the tongue deformation, by the measurement of changes in the time of flight (ToF) of ultrasound echoes reflecting back from the tongue-food interface. This type of mono-element transducer thus allows a precise quantification of the deformations in the direction corresponding to the beam axis. However, they only reflect what happens in the core of the samples (with variable beam sizes, but generally several millimeters in diameter), and they are thus not as suitable as multi element array probes for quantifying gradients of deformation along the direction perpendicular to the beam (from the core to the periphery of the interfaces).

Against this backdrop, the aim of the study presented here was twofold. First, it sought to build upon previous research by determining whether a quantitative ultrasound method could characterize not only the deformation of an artificial tongue, but also that of a heterogeneous food, modelled using stacked gels. Second, it explored how the deformation dynamics of the system and its components were influenced by (i) associations between gel layers; (ii) differences in mechanical properties between the two layers; and (iii) layer position on the artificial tongue. Drawing upon the work of Santagiuliana et al. (2018), agar and gelatin gels of differing concentrations (which thus displayed different mechanical properties) were created and combined to form stacked gels consisting of two layers. Then, the gels were placed on a deformable artificial tongue model (ATM), and the entire system underwent uniaxial compression induced by a hard, flat probe that mimicked the palate (Figure 4.1a). Ultrasound pulse-echo approaches and associated signal processing methods were developed to monitor the deformation dynamics of the different system components during the compression test.

## 4.2. Materials and methods

### 4.2.1. Preparation of the stacked gels

Two types of polymers were used in this study: agar (Ag) and gelatin (Ge). Gel composition and preparation protocols were similar to those in Santagiuliana et al. (2018). Each gel layer was identified using a polymer abbreviation (Ag and Ge) followed by a number corresponding to the gel's fracture stress (kPa). The solutions of agar (HP700IFG, Kalys, Bernin, France) and gelatin (Bloom 250 PS 8/3 Rousselot, Gent, Belgium) were prepared using demineralized water and then stirred for 30 min to allow for hydration. Then, the solutions (inside capped bottles) were heated in a water bath under continuous stirring conditions. The heating regime differed for the two polymer types: 45 min at 95 °C for the agar, and 20 min at 60 °C for the gelatin. The solutions were subsequently poured into cylindrical syringes (diameter: 26 mm, volume: 65 mL; Omnifix, B. Braun); the syringes' interiors had been lightly coated with sunflower oil. The syringes were kept at 4 °C for 15–18 h to allow gelation to occur. Afterwards, the cylindrical gels were removed from the syringe using the plunger and cut into 5-mm-thick cylinders using a custom-built cutting frame. The gels were then assembled by stacking two individual cylinders. The two stacked layers were not glued with any external adhesive agent. Combinations were created with cylinders that differed in polymer type and fracture stress (Figure 4.1b). To improve the echogenic properties of the interface between the two layers, the top of the lower layer was dusted with a small quantity (< 10 mg) of agar powder prior to the placement of the upper layer. Preliminary experiments confirmed that the powder did not have any effects on the mechanical properties of the stacked gels.

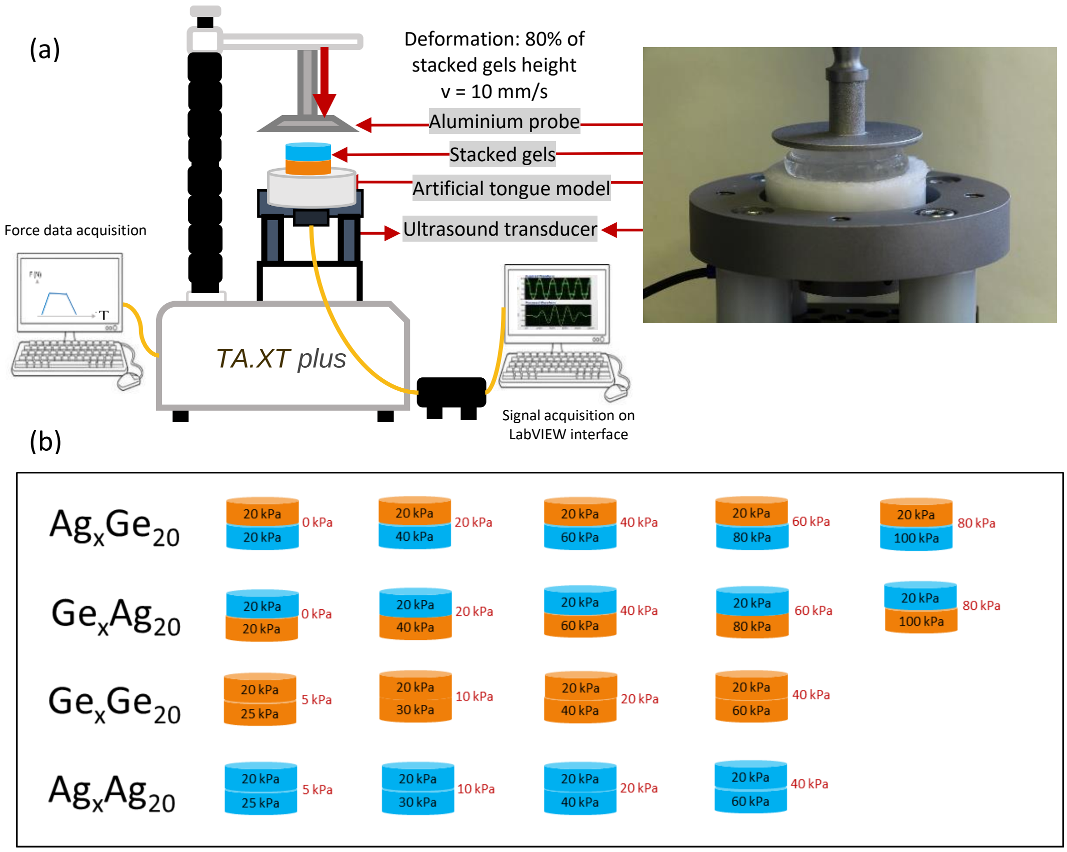



**Figure 4.1: (a) Schematic representation of the experimental set-up; (b) the combinations of stacked gels tested (including the flipped combinations).**

### 4.2.2. Preparation of the artificial tongue model

The ATM employed in this study was also used in Srivastava et al. (2021). To describe the preparation method briefly, a polyvinyl alcohol solution (PVA; Sigma Aldrich, Saint-Louis, USA) was made using ultra-pure water (10% w/w); it then underwent stirring at 80 °C for 2 h. The solution was subsequently cooled to 20 °C and poured into a cylindrical mold made of polyvinyl chloride (diameter: 50 mm, height: 20 mm); at the bottom of the mold was P40 sandpaper (grain size: 425 µm; Norton, Saint-Gobain, France). The sandpaper was used to create a topological surface profile similar to that of a human tongue. After being carefully sealed, the mold and its contents were frozen (-20 °C for 10 h) and subsequently thawed (20 °C for 14 h). A total of six freezing-thawing cycles were performed to obtain the proper rigidity for a PVA hydrogel (Fromageau et al., 2007). Finally, the ATM was unmolded and stored in reverse osmosis treated water at room temperature (20 °C). This PVA based ATM can be stored at room temperature without any deterioration and any modification of mechanical properties for several months.

Young's modulus and surface roughness values for the ATM had been previously characterized using a texture analyzer and a contact profilometer, respectively (Srivastava et al., 2021). The Young's modulus value was 71 ± 2 kPa. Ishihara et al. (2013) found that the rigidity values of the human tongue at rest and in a contracted state were 12.2 ± 4.2 kPa and 122.5 ± 58.5 kPa, respectively. Surface roughness profile in terms average peak height ($R_a$) and correlation length (peak width, β) was 103 ± 3 µm and 429 ± 63 µm respectively. For the human tongue, average peak height appears to be 42.5–101.4 µm (Uemori, Kakinoki, Karaki, & Kakigawa, 2012), while peak width appears to be 355–878 µm (Andablo-Reyes et al., 2020).

### 4.2.3. Test sequence and ultrasound measurements

The ultrasonic analysis equipment and measurement principles are very similar to what was done in our previous studies, in which the analysis technique and procedure are also described in detail (Mantelet et al., 2020a; Mantelet et al., 2020b; Srivastava et al., 2021). The main changes are the choice of a higher frequency ultrasound transducer, the use of a US pulser-receiver with a higher pulse recurrence frequency, and the development of a new image processing method for the analysis of the different interfaces. All these new elements are described in detail in the following lines.

The experimental set-up (Figure 4.1a) included a texture analyzer, an ATM and a 5 MH mono-element piezoelectric ultrasound transducer (Olympus, Shinjuku, Tokyo, Japan) placed in contact (underneath) with the ATM. The use of a 5-MHz ultrasound frequency resulted in improved spatial resolution compared to previous work (maximum ultrasound frequency of 1 MHz; Mantelet et al., 2020a and b), which was crucial for performing measurements at the tight interface between the two layers. The transducer was connected to an ultrasound pulser-receiver (Ultrasound-Key, Lecoeur Electronique, Chuelles, France), which produced a negative square pulse wave signal (width setting: 24/255, amplitude: 100 V). The transducer digitized the radio frequency (*rf*) signals corresponding to the pulse-echo response of the system (quantification: 12 bits, sampling rate: 80 MHz, gain: 20 dB). The acquisition of the rf signals was done in real time during the tests using a dedicated user interface developed with LabVIEW® (LabVIEW, National Instruments, Austin, Texas, USA). The pulse recurrence frequency was approximately 450 Hz. This level was significantly higher than that used in previous research (maximum of 90 Hz; Mantelet et al., 2020a and b), allowing for better temporal resolution during the fast processes taking place. As a result, it was possible to conduct more detailed analyses of the signal fluctuations associated with the mechanical phenomena occurring at the interface between layers.

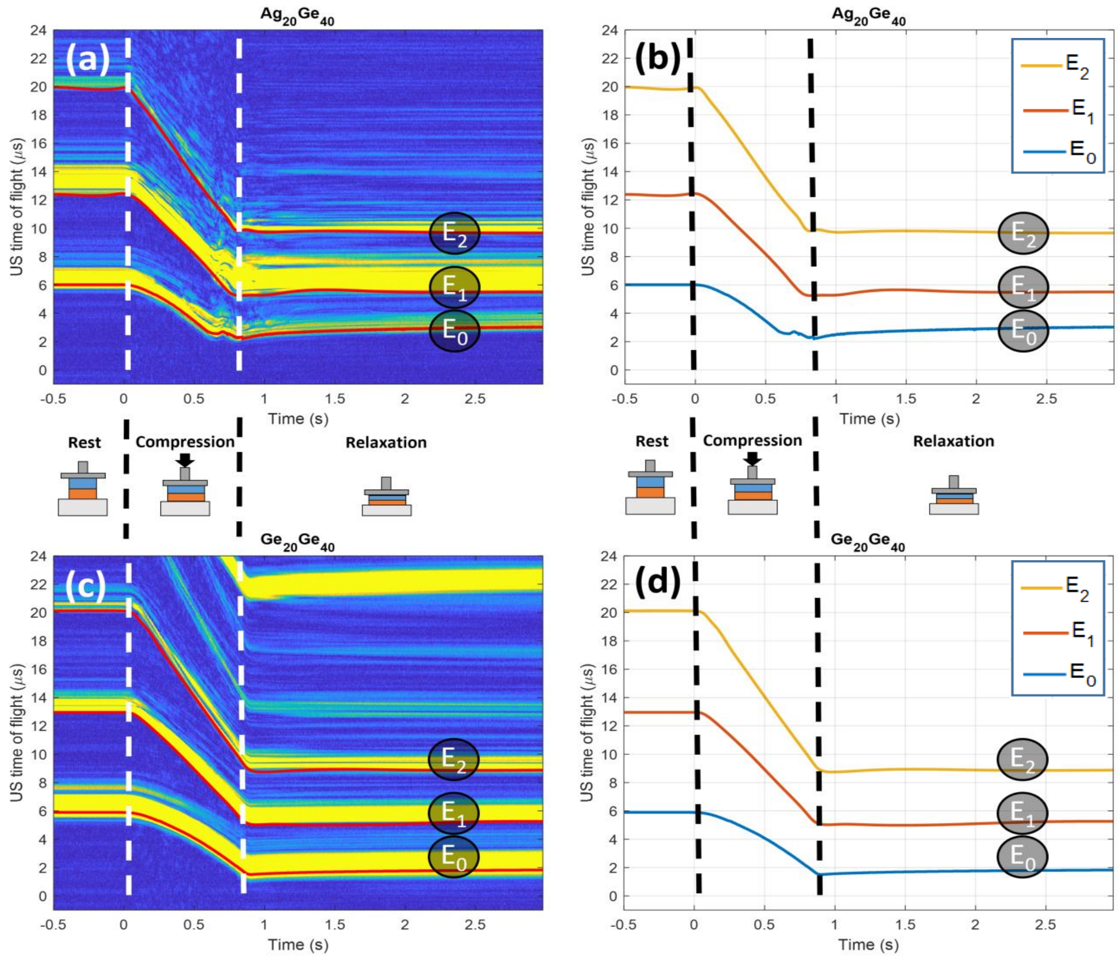
Before beginning the compression test, any water on the ATM's surface was carefully removed using absorbent paper (Kimtech$^{TM}$, Kimberly-Clark, Irving, TX, USA). A stacked gel combination was then gently placed on the ATM. The circular aluminum probe (diameter: 40 mm) of the texture analyzer was used to apply uniaxial stress to the gel-ATM system. The amplitude of the probe displacement was 80% of the thickness of the stacked gels, subjecting the entire system (ATM [20 mm] and the two gel layers [5 mm each]) to compression, imposing a deformation rate of approximately 25% (when considering the ATM and the gel as a single entity). Compression speed was 10 mm/s, and the system was kept in a compressed state for 2.5 seconds before the probe was lifted. This holding step was necessary to examine relaxation phenomena. The probe is not lifted up during the latter stage.

During each test, the ultrasound measurements were performed in two steps. First, a reference signal was acquired by putting the transducer in contact with the ATM prior to gel placement. Then, ultrasound signals were acquired in real time (approximately 450 signals per second) during the test sequence, which began 0.5 s before compression was initiated and was followed by the 2.5-s holding step. It is also important to highlight that the ultrasonic system has negligible thermal effects due to the low intensity and short duration of the pulses.

**4.2.4. Signal analysis**

High-frequency noise reduction with a low-pass filter (cut-off frequency: 8 MHz) was applied to the fast Fourier transforms (FFTs) of all the *rf* signals. Figure 4.2a and 4.2c show a motion-mode (M-mode) image of the modulus of the Hilbert transform of all the signals acquired over the course of two tests, viz. $Ag_{20}Ge_{40}$ and $Ge_{20}Ge_{40}$ respectively. M-mode images depict changes in ultrasound signals over time. The x-axis corresponds to the time elapsed during a test. The y-axis corresponds to the time elapsed after each ultrasonic signal has been emitted. At a rate of approximately 450 *rf* signals were emitted per second. In Figure 4.2a and 4.2c, there are three yellow traces—each corresponds to an echo generated by an ultrasound wave meeting an interface. $E_0$ corresponds to the acoustic energy reflected from the interface between the ATM and the lower gel layer (layer #1). $E_1$ is the response for the interface between the lower and upper gel layers (layers #1 and #2). $E_2$ is the response for the interface between the upper food layer and the probe. During a test sequence, all the echoes evolved, both in terms of amplitude (peak height) and of ToF (peak time of occurrence).

In the first phase of signal processing, an analysis was performed of ToF_$E_0$, the time of flight of the echo $E_0$. The ToF calculation determined the first zero-crossing (i.e., the time point where the sign of *rf* signal amplitude changes) after the signal amplitude had exceeded a threshold equal to twice the level of noise. This process yielded ToF_$E_0$(t) for the entire experimental test (Figure 4.2b and d).




**<u>Figure 4.2:</u> Ultrasound M-mode image, where the x-axis is the time elapsed during a given test and the y-axis is the time delay between ultrasound emission and echo detection by the transducer (a: $Ag_{20}Ge_{40}$ and c: $Ge_{20}Ge_{40}$); variation in time of flight for the $E_0$, $E_1$, and $E_2$ echoes during a test, obtained from the analysis of the M-mode image and the corresponding *rf* signals (b: $Ag_{20}Ge_{40}$ and d: $Ge_{20}Ge_{40}$).**

In the second phase of signal processing, an analysis was performed of the ToF of the echoes $E_1$ and $E_2$ (ToF_$E_1$ and ToF_$E_2$, respectively). Semi-automatic procedures were developed to perform this analysis due to the complexity of the ultrasound signals that followed echo $E_0$ (multiple reflections, overlapping signals, highly variable amplitude). First, the M-mode image for the entire trial was plotted. The modulus of the Hilbert transform of the signals was used (as in Figure 4.2a and c). As mentioned above, such plots allow all the echoes from an experimental trial to be clearly visualized. Next, a set of points in the profiles of $E_1$ and $E_2$ was manually selected. Due to the duration of the ultrasound pulse, the yellow traces corresponding to each echo had a specific width in the M-mode image (see Figure 4.2a and c). When selecting the points, the idea was to follow the changes in each echo over time, moving from the blue zone (before the echo occurred) to the yellow zone (at the beginning of the echo). This method allowed ToF values to be selected in relation to the first zero-crossing event. Then, a MATLAB program employing an interpolation function (Spline method: cubic interpolation of values at neighboring points) was utilized to generate the full profiles of changes in ToF for each echo over the course of the experimental trial (ToF_$E_1$[t] and ToF_$E_2$[t] for $E_1$ and $E_2$, respectively; Figure 4.2b and 2d).

### 4.2.5. Data Processing

Signal processing yielded ToF data for the echoes originating from the three interfaces: ToF_$E_0$(t), ToF_$E_1$(t), and ToF_$E_2$(t). During a given test, there were three time points of interest—$t_0$, $t_1$, and $t_2$—that corresponded to the moment before compression, the end of compression step, and the end of 2.5 s holding step, respectively. During each test, the change in ToF for each echo depended on two main parameters. The first parameter was the propagation speed of the ultrasound waves, which was similar for the ATM, agar gel, and gelatin gel (~1,500 m/s). It varied a negligible amount during compression, as it is governed above all by media compressibility and density (which were highly similar in this study). The second parameter was the path length of the ultrasound wave. The greater the length, the longer the flight time. In the experimental tests, the path length of the wave within one of the layers (ATM, agar gel, or gelatin gel) was twice as long as layer thickness. The deformation of each system component can thus be estimated as follows:

ATM deformation in millimeters ($\Delta l_T$):

$$\Delta l_T(t) = \frac{1500}{2}\left(TOF_{E_0}(t) - TOF\_E_0(t_0)\right) \quad (1)$$

Layer 1 deformation in millimeters ($\Delta l_{L1}$):

$$\Delta l_{L1}(t) = \frac{1500}{2}\left(TOF_{E_1}(t) - TOF\_E_1(t_0)\right) - \Delta l\varepsilon_T(t) \quad (2)$$

Layer 2 deformation in millimeters ($\Delta l_{L2}$):

$$\Delta l_{L2}(t) = \frac{1500}{2}\left(TOF_{E_2}(t) - TOF\_E_2(t_0)\right) - \Delta l_{L1}(t) \quad (3)$$

This should be noted that the radius of the ultrasound beam (around 10 mm) is less than that of the gel layer. Hence the obtained values by above equations only indicates deformations observed at very central part of ATM-gel interface.

The resulting values of $\Delta l_T$, $\Delta l_{L1}$, and $\Delta l_{L2}$ thus convey the real-time changes in thickness for each component. Another approach is to express these values relative to the global deformation applied to the system (which also corresponds to the amplitude of probe displacement). These percentages can be used to analyze the relative deformation experienced by each system component:

$$\varepsilon_T(t) = 100 * \frac{\Delta l_T(t)}{\Delta l_T(t)+\Delta l_{L1}(t)+\Delta l_{L2}(t)} \quad (4)$$

$$\varepsilon_{L1}(t) = 100 * \frac{\Delta l_{L1}(t)}{\Delta l_T(t)+\Delta l_{L1}(t)+\Delta l_{L2}(t)} \quad (5)$$

$$\varepsilon_{L2}(t) = 100 * \frac{\Delta l_{L2}(t)}{\Delta l_T(t)+\Delta l_{L1}(t)+\Delta l_{L2}(t)} \quad (6)$$

It is important to note that these indicators do not correspond to the deformation endured by the layers in relation to their initial thickness. It is important to note that these indicators do not correspond to the deformation endured by the layers in relation to their initial thickness.

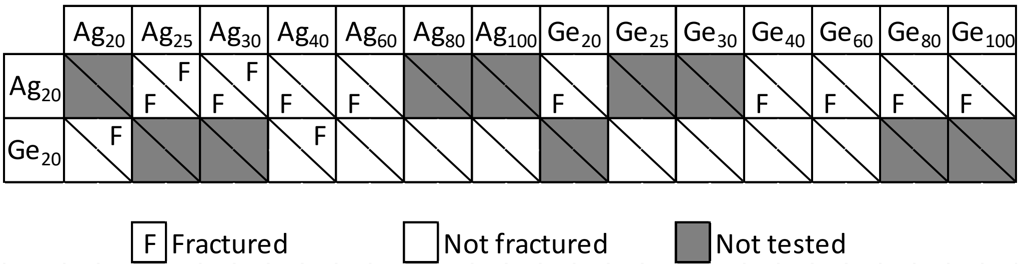
In this study, the obtained results of deformations (for a set of at least seven repetitions) are presented and analyzed in different ways. Some parameters (force and ATM deformation) are studied by a temporal evolution throughout a test. For those, a graphical representation in the form of an average curve and an envelope corresponding to the standard deviation was chosen. This type of representation allows discussion of several conditions on the same graph. And, to conclude on the significance of differences, the envelopes around the curves were taken into account.

The second type of data (distribution of deformation in the individual layers) representation and analysis is done at given time spots ($t_0$, $t_1$ and $t_2$) during a test. For this, a representation in the form of histograms was used where each bar corresponds to a mean value, and the error bar to a standard deviation. Here, an ANOVA test was performed using XLSTAT (Addinsoft, Paris, France) with a complimentary post hoc analysis (Tukey HSD) test.

## 4.3. Results and discussion

The different stacked gels considered in the present study are summarized in Table 1 along with information on the observation of the fracture within each monolayer layer during the tests. The mechanical properties of all the monolayer gels used in this study (to build different combinations) had been previously characterized by Santagiuliana et al. (2018) — these values of Young's modulus and fracture stress are republished here in Table 2. [The formulation of the gels were done strictly as in aforesaid study to obtain the same mechanical values.] However, for fracture strain of each monolayer, the values reported in the aforesaid publication have here been converted to be expressed as a percentage of total displacement of the probe during our acoustical experiments, where a total deformation of 8 mm was applied on the system comprising three components (30 mm thick): a ATM (20 mm thick) and two layers of food gels (each 5 mm thick). The values of fracture strain described in the table thus correspond to the proportion of the total displacement of the plate from which the fracture of a 5 mm thick gel is expected. This was done to facilitate easier discussion with the parameters $\varepsilon_{L1}$ and $\varepsilon_{L2}$. It can be noted that the agar and gelatin monolayer gels have distinctly different fracture strains but within themselves they didn't vary greatly (agar: 21.0–23.4% and gelatin: 39.3–46.9%). The differences in the obtained fracture strain between gelatin and agar gels is primarily due to the polymer type and resulting gel microstructure. Ikeda, Sangu, & Nishinari (2003) have shown that agar gels show lower fracture strain values when compared to gelatin. Also the study further reports that with increase in polymer concentration gelatin based gels have shown increments in fracture strain while a slight decrease was seen for agar based gels. Finally, with regard to Young's modulus, it can be seen that agar monolayer gels had much varied values than gelatin gels (agar: 11.3–125.8 kPa and gelatin: 8.0–40.7 kPa).

**Table 4.1: Tabular representation of the different combinations of gel layers selected to form the stacked gels considered in the present study (in white). The presence of the letter "F" in a box indicates that the corresponding layer fractured during the experiments (confirmed both by visual inspection of the gels after the tests and by the presence of sudden drops in the ultrasound traces).**



**<u>Table 4.2:</u> Mechanical characteristics—Young's modulus, fracture stress, and fracture strain values—of different individual gels (reproduced from Santagiuliana et al., 2018). The means and standard deviations (SD) are indicated. Threshold fracture strain (%) was calculated with respect to the total deformation of the food-tongue system (percentage of the total amplitude of probe displacement [8 mm] at which a 5-mm-thick gel is expected to fracture).**

| Gel | Polymer concentration (w/w in water) | Young's modulus (kPa) | | Fracture stress (kPa) | | Fracture strain (%) | |
|---|---|---|---|---|---|---|---|
| | | Mean | SD | Mean | SD | Mean | SD |
| $Ag_{20}$ | 0.81 | 11.3 | 1.7 | 19.7 | 1.2 | 21.0 | 1.2 |
| $Ag_{25}$ | 0.94 | 14.2 | 3.3 | 24.7 | 1 | 21.4 | 1.2 |
| $Ag_{30}$ | 1.06 | 18.7 | 2.7 | 30.6 | 1 | 22.6 | 1.2 |
| $Ag_{40}$ | 1.31 | 24.7 | 6 | 39.8 | 2 | 23.4 | 1.2 |
| $Ag_{60}$ | 1.81 | 48.3 | 8 | 62.1 | 2.7 | 23.4 | 1.2 |
| $Ag_{80}$ | 2.31 | 67.1 | 20.5 | 79.6 | 3.5 | 23.4 | 1.8 |
| $Ag_{100}$ | 2.80 | 125.8 | 12 | 102.1 | 3.2 | 23.0 | 0.6 |
| $Ge_{20}$ | 4.03 | 8 | 1.6 | 20.5 | 2.5 | 39.3 | 4.8 |
| $Ge_{25}$ | 4.54 | 10.3 | 2.1 | 25.2 | 1.3 | 39.5 | 3.0 |
| $Ge_{30}$ | 5.05 | 11.7 | 2 | 31.3 | 2.7 | 40.8 | 5.4 |
| $Ge_{40}$ | 6.07 | 18.5 | 4.3 | 40.9 | 2.7 | 42.1 | 4.8 |
| $Ge_{60}$ | 8.11 | 24 | 3.2 | 60.7 | 3.5 | 43.7 | 5.4 |
| $Ge_{80}$ | 10.15 | 33.3 | 5.2 | 78.8 | 3.3 | 44.6 | 4.2 |
| $Ge_{100}$ | 12.19 | 40.7 | 4.3 | 103.4 | 3.7 | 46.9 | 7.1 |

In this section, first we discuss the impact of the overall mechanical properties of the gels on the ultrasound-assisted measurement of ATM deformations. Then, the focus is placed on characterization of the individual behaviors of the different layers composing the stacked gels. In each of these two parts, we will thus be able to better understand the impact of different parameters on the mechanical behavior of stacked gels subjected to compression and then to relaxation, between a deformable ATM and a hard plate. These behaviors will be studied by considering several factors: impact of the type of polymer association, the polymer content of the gels, or the orientation of the gels (surface in contact with the ATM). It is important to recall that, in the study's experimental set-up, the diameter of the ultrasound beam (less than 10 mm) was smaller than the diameter of the gels (26 mm prior to compression).

### 4.3.1. Impact of gel properties on the deformation of the artificial tongue model

First, the effect of gels properties on ATM deformation was examined. The ultrasound measurements made it possible to correlate the degree of deformation with the force measured by the texture analyzer. This relationship is important because, in real life, tongue deformation during food oral processing could directly impact mechanoreceptor stimulation. In particular, two experimental situations were considered: tests in which a $Ge_{20}$ layer was coupled with a variety of gelatin layers ($Ge_{25}$, $Ge_{30}$, $Ge_{40}$, and $Ge_{60}$; Figures 4.3a and b) and tests in which an $Ag_{20}$ layer was coupled with a variety of agar layers ($Ag_{25}$, $Ag_{30}$, $Ag_{40}$, and $Ag_{60}$; Figures 4.3c and d). Figure 4.3a presents the variation of force exerted during the compression of stacked gels both composed of gelatin, with top layer as $Ge_{20}$, combined with bottom layers with varied fracture stresses ($Ge_{25}$, $Ge_{30}$, $Ge_{40}$, $Ge_{60}$). The envelopes around bold colored lines represent standard deviation obtained over seven repetitions. Figure 4.3b represents, for the same gels, the evolution of the ATM deformation over the course of compression. It can be observed from the figures that the variation of estimated ATM deformation was in accordance with that of force, which is congruent with the observation from our previous results (Srivastava et al., 2021). However, varying the fracture stress of the lower gelatin layers paired with $Ge_{20}$ had no significant impact (overlapping standard deviation envelopes), neither at the end of compression step or during relaxation.

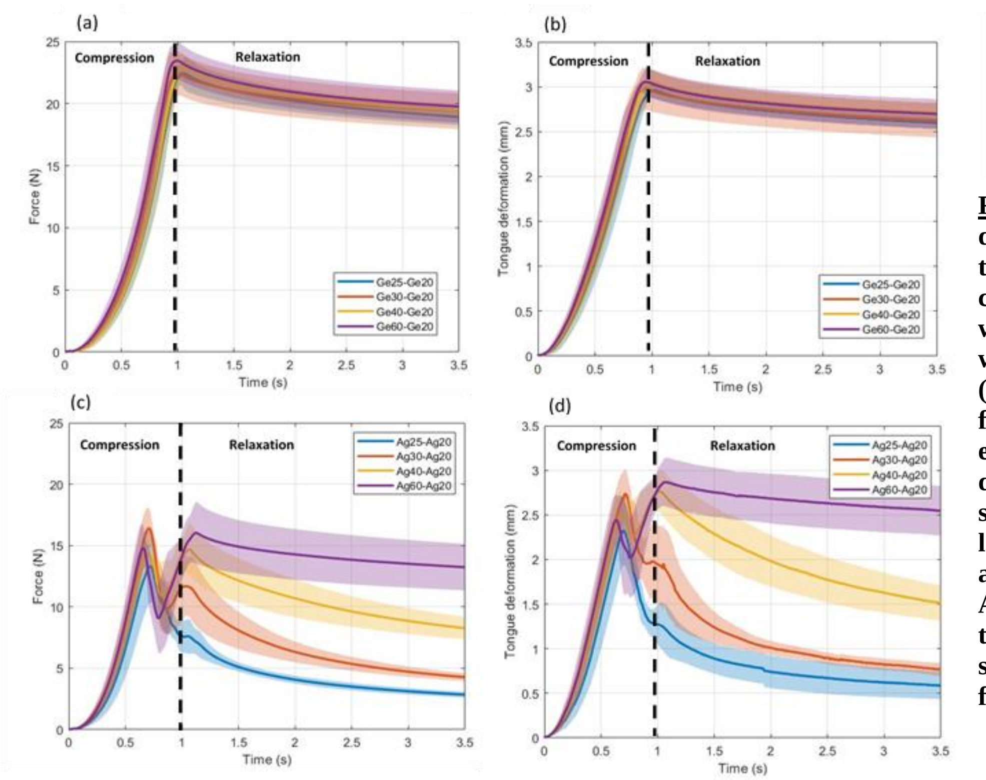
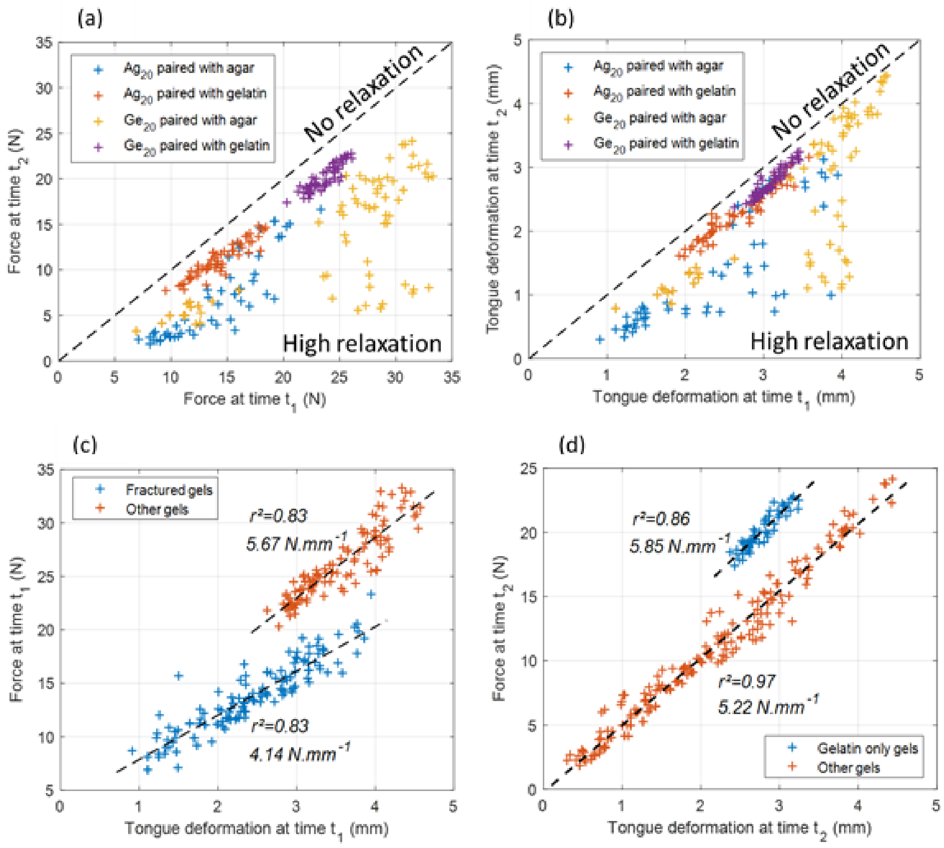



**Figure 4.3: (a) Force and (b) deformation experienced by the ATM during the compression of stacked gels in which a $Ge_{20}$ layer was paired with another gelatin layer ($Ge_{25}$, $Ge_{30}$, $Ge_{40}$, or $Ge_{60}$); (c) force and (d) deformation experienced by the ATM during the compression of stacked gels in which an $Ag_{20}$ layer was paired with another agar layer ($Ag_{25}$, $Ag_{30}$, $Ag_{40}$, or $Ag_{60}$). The envelopes around the thick colored lines show the standard deviations obtained from seven replicates.**

In the second situation, where $Ag_{20}$ served as the upper layer and the second agar layer was on bottom (Figure 4.3c and d), varied responses were obtained. During compression (between 0 and 1 s), the sudden drop in force signaled that at least one of the gel layers had fractured. The results also indicate that ATM deformation varied markedly during the relaxation. In real life, such phenomena could contribute to perceived heterogeneity and should hence be studied further. At the end of 2.5 s holding step, different stacked gels displayed greatly contrasting force and deformation profiles. The higher the agar concentration in the lower layer (i.e., the higher the fracture stress), the smaller the decrease in the force and deformation experienced by the ATM. In all cases, the upper $Ag_{20}$ layer fractured (Table 1); however, lower layers when composed of $Ag_{40}$ and $Ag_{60}$ did not. Consequently, comparing the results for the gelatin and agar gels, the brittle nature of agar might explain the drastic variation in force and deformation that was observed. Furthermore, the sudden appearance of variation in deformation was consistent with the visual signs of fracture in the samples at the end of the tests (Table 1).

For all the gel combinations used in the study, an analysis was performed of force patterns at the end of compression step ($t_1$) and holding step ($t_2$) (Figure 4.4a); ATM deformation at these two time points was also examined (Figure 4.4b). Measurements were organized into four different groups based on the four biopolymer combinations studied. In both figures, a dotted line indicates where force and deformation were identical at times $t_1$ and $t_2$. Thus, when a point is closer to this line (i.e., "no relaxation"), it indicates that there was less change in the force and deformation experienced by the ATM during relaxation. Conversely, when a point is closer to the x-axis (i.e., "high relaxation"), it indicates that the force and deformation experienced by the ATM decreased during relaxation. The results highlight the similarities in the distribution of points within the different groups (Figure 4.4a and b).

When $Ag_{20}$ or $Ge_{20}$ were paired with different gelatin layers, profiles of force and deformation for the ATM were fairly similar across gels combinations (especially when both layers were gelatin based) (Figure 4.4a and b; orange and purple points). This finding can be explained by the fact that gelatin gels are more elastic. When the stacked gels were entirely composed of gelatin, the measurement points clustered together, suggesting a consistent response despite the differences in Young's modulus values. They displayed a low degree of relaxation behavior, given that they remained close to the "no relaxation" line. The reason behind the low relaxation in gelatin based gels could be due its prominent strain hardening behavior at higher deformation.




**<u>Figure 4.4:</u> (a) Force experienced by the ATM at the end of holding step ($t_2$) versus at the end of compression step ($t_1$); (b) deformation experienced by the ATM at the end of holding step ($t_2$) versus at the end of compression step ($t_1$); (c) force versus deformation at the end of compression step ($t_1$); (d) force versus deformation at the end of holding step ($t_2$).**

However, when a layer composed of $Ag_{20}$ or $Ge_{20}$ was paired with different layers of agar, there was greater variability in these profiles at the end of compression step, and, comparatively, a higher degree of relaxation was observed (Figure 4.4a and b; blue and yellow points). The measurement points were more scattered because of the higher fracture behaviors reported in the agar-agar combinations due to higher stress concentration. Moreover, few of the agar layers also fractured even when paired with a gelatin layer (Table 1).

Although the force and deformation experienced by the ATM was generally consistent across groups (Figure 4.4a and b), slight differences were observed. For example, the two point clusters (blue and yellow) corresponding to stacked gels with an $Ag_{20}$ or $Ge_{20}$ layer paired with agar layers displayed distinct force values (Figure 4.4a); there was some overlap, however, in deformation values (Figure 4.4b). Moreover, when $Ge_{20}$ was paired with agar, some of the data points in the high range of deformation at time $t_1$ (between 3.5 and 5 mm) exhibited low relaxation behavior in terms of ATM deformation (remaining close to the dotted line) which could not be confirmed on the force plots.

The next step was to compare the force and deformation experienced by the ATM at the end of compression step ($t_1$) and holding step ($t_2$) (Figures 4.4c and d, respectively). Two distinct clusters of points can be seen; each seems to form a straight line. It appears that the clusters correspond to, respectively, the stacked gels in which compression caused at least partial fracturing ($r^2$=0.83) versus no fracturing ($r^2$=0.83) (Figure 4.4c). Cluster composition however differed at time $t_2$ (Figure 4.4d). One cluster consisted of the 100% gelatin stacked gels ($r^2$=0.86), while the other consisted of all the other combinations, meaning gels with at least one agar layer ($r^2$=0.97).

In figure 4.4c, for the same level of deformation, it can be seen that, at the end of compression step, higher levels of force were associated with gels that had not fractured. One possible explanation for the observed differences is that the surface upon which the force was exerted was not the same. Consequently, for a given level of deformation, the higher force values could have been associated with larger contact surfaces. The differences in surface characteristics between the ATM and the gel could explain these different behaviors. During compression, adhesion and friction between the bottom-layer gel and the ATM may have acted in an opposing manner to the increase in contact area (i.e., a barreling effect; Brennan & Bourne, 1994; Pons & Fiszman, 1996). Under conditions of compression, a barreling effect can arise due to friction between a cylindrical sample and the plate below (in this study, an ATM), leading to triaxial instead of uniaxial stress. Experiencing deformation, the gel may thus have tended to bend even as the area in contact with the ATM remained the same. A similar pattern emerged at the end of holding step for the 100% gelatin stacked gels, as compared to the other gel combinations (Figure 4.4d). The non-linear response of gelatin gels under high deformations (i.e. strain hardening) when compared to more linear response of agar gels until fracture (due to higher brittleness) could explain the higher force for gelatin only gels at relaxation step.

It is important to recall that, in the study's experimental set-up, the diameter of the ultrasound beam (less than 10 mm) was smaller than the diameter of the gels (26 mm prior to compression). Thus, the deformation measured by ultrasound only reflected what was happening in the central part of the ATM. To obtain information about radial ATM deformation, it would be necessary to use a multiple-element transducer (an array-type transducer, as employed in imaging). It would then be easier to observe any barreling effects.

The results discussed in this section underscore the utility of ultrasound methods, highlighting their complementarity with force measurement methods. The latter make it possible to study the mechanical responses of multi-layer heterogeneous materials, such as an ATM in contact with stacked gels differing in mechanical properties. However, the results also made it clear that a comprehensive mechanistic interpretation of our findings requires characterizing the phenomena that occurred within the stacked gels. The second part of the discussion focuses on this subject.

**4.3.2. Distribution of deformation: the tongue-food system**

This study makes a contribution to the field by describing an ultrasound method for investigating how the different components of a model tongue-food system experience deformation over time. Thanks to this methodology, it was clear that deformation dynamics varied across experimental situations (Figure 4.5). First, results were analyzed for the stacked gels in which the upper layer was $Ag_{20}$ and the lower layer was also an agar gel ($Ag_{25}$, $Ag_{30}$, $Ag_{40}$, or $Ag_{60}$); the deformation of the ATM ($\varepsilon_T$), lower layer ($\varepsilon_{L1}$), and upper layer ($\varepsilon_{L2}$) were examined relative to the total deformation experienced by the system. For example, under conditions of compression, 17% of the total deformation was shared by the ATM, 36% by the $Ag_{25}$ gel, and 46% by the $Ag_{20}$ gel (first bar in Figure 4.5a).

Two other factors affected the results: layer position and the time at which the measurement was made (compression vs. relaxation) (Figure 4.5). In one situation, $Ag_{20}$ served as the upper layer (Figures 4.5a and b), while in another situation, the sample was flipped, and $Ag_{20}$ served as the lower layer (Figures 4.5c and d). When $Ag_{20}$ was on top, the system underwent compression (Figure 4.5a: measurements at $t_1$) and relaxation (Figure 4.5b: measurements at $t_2$). The same was true when $Ag_{20}$ was on the bottom (Figure 4.5c: measurements at $t_1$ and Figure 4.5d: measurements at $t_2$).

When $Ag_{20}$ served as the upper layer, it experienced high and consistent levels of deformation that were independent of the composition of the lower agar layer (Figure 4.5a). These levels (46–48%) were much greater than the fracture threshold (21.0% on average; Table 2); this result was consistent with observed fracturing at the end of the tests and or when tracing the ToF profiles. As the concentration of agar in the lower layer climbed ($Ag_{25}$, $Ag_{30}$, $Ag_{40}$, and $Ag_{60}$), there was a clear trend in which the increase in Young's modulus values led to the decreased deformation of the lower agar layers and the increased deformation of the ATM (i.e., in compensation). The deformation levels of $Ag_{25}$ and $Ag_{30}$ were consistent with the visual signs of fracturing. Under conditions of relaxation (Figure 4.5b), the $Ag_{20}$ gel on top continued to undergo deformation, while the ATM returned to its initial state. A degree of compensation was provided by the fractured lower agar layer. Such was also true when $Ag_{25}$ and $Ag_{30}$ were used in the bottom layer—they continued to experience deformation during the relaxation stage. The same general trends were seen when the stacked gels were flipped and the $Ag_{20}$ layer was found on bottom (Figures 4.5c and d). Although the differences were not significant (based on the overlap in the standard deviations), the $Ag_{20}$ layer tended to experience less deformation when it was on bottom. In contrast, the deformation of the upper layer became more pronounced. When the upper layer was composed of $Ag_{40}$, the level of deformation (40%) was much higher (Figure 4.5d) than in all the other cases; it was also higher than the fracture threshold (23.4%; Table 2), suggesting that fracture was very likely to occur. These differences resulting from layer position could be explained by changes in surface interactions, namely whether gel layers were in contact with the ATM (composed of soft PVA) or with the palate-mimicking probe (composed of rigid aluminum). These interactions may promote or mitigate gel spreading over the course of compression or relaxation. In summary, the results demonstrate that the ultrasound method did a good job of describing the deformation dynamics of the system's individual parts. The fact that slight differences were also observed when the stacked gels were flipped upside down is encouraging because it reflects the method's ability to independently distinguish deformation in the independent layers.

An analysis was then performed of the deformation dynamics in situations where a $Ge_{20}$ layer was coupled with different types of agar layers (Figure 4.6). The experimental approach was the same as that described above. In one situation, the upper layer was composed of $Ge_{20}$ (Figures 4.6a and b), while in another situation, the sample was flipped, and the lower layer was composed of $Ge_{20}$ (Figures 4.6c and d). When $Ge_{20}$ was on top, the system experienced compression (Figure 4.6a: measurements at $t_1$) and relaxation (Figure 4.6b: measurements at $t_2$). The same was true when $Ge_{20}$ was on the bottom (Figure 4.6c: measurements at $t_1$ and Figure 4.6d: measurements at $t_2$). The average deformation values for the $Ge_{20}$ layer ranged between 26 and 40%, remaining, in the vast majority of cases, below the fracture threshold (39.3%; Table 2). These measurements are thus consistent with the observation that the $Ge_{20}$ layer was never fractured at the end of a test. Layer position generally had no effect on how the stacked gels responded to compression (Figures 4.6a and c). However, the case in which a $Ge_{20}$ layer was paired with an $Ag_{20}$ layer was distinct. As noted above, the $Ag_{20}$ layer experienced significant deformation (43–48%, above the fracture threshold). Conversely, when a $Ge_{20}$ layer was paired with an agar layer composed of $Ag_{40}$, $Ag_{60}$, $Ag_{80}$, or $Ag_{100}$, the deformation of the agar gels was less pronounced and remained below the fracture threshold. Interestingly, even though these agar gels represented a wide range of Young's modulus values (24.7–125.8 kPa), they differed little in their deformation distributions. However, more marked differences appeared during relaxation (Figures 4.6b and d). It should also be noted that these pronounced differences in the deformation of agar layer at $t_1$ and $t_2$ reflects the possibility of fracture to take place after the compression step. In particular, the deformation of the Ag40 layer exceeded the fracture threshold. Similarly, the deformation of the $Ag_{60}$ layer tended to increase as well. As agar concentration climbed, moving from $Ag_{20}$ to $Ag_{100}$, it was observed also that the deformation profile of the ATM changed significantly in the seconds following compression. Therefore, when the palate-mimicking probe was held in compression position, prominent mechanical phenomena (including fractures) occurred and could be monitored with ultrasound over response times longer than the duration of compression.

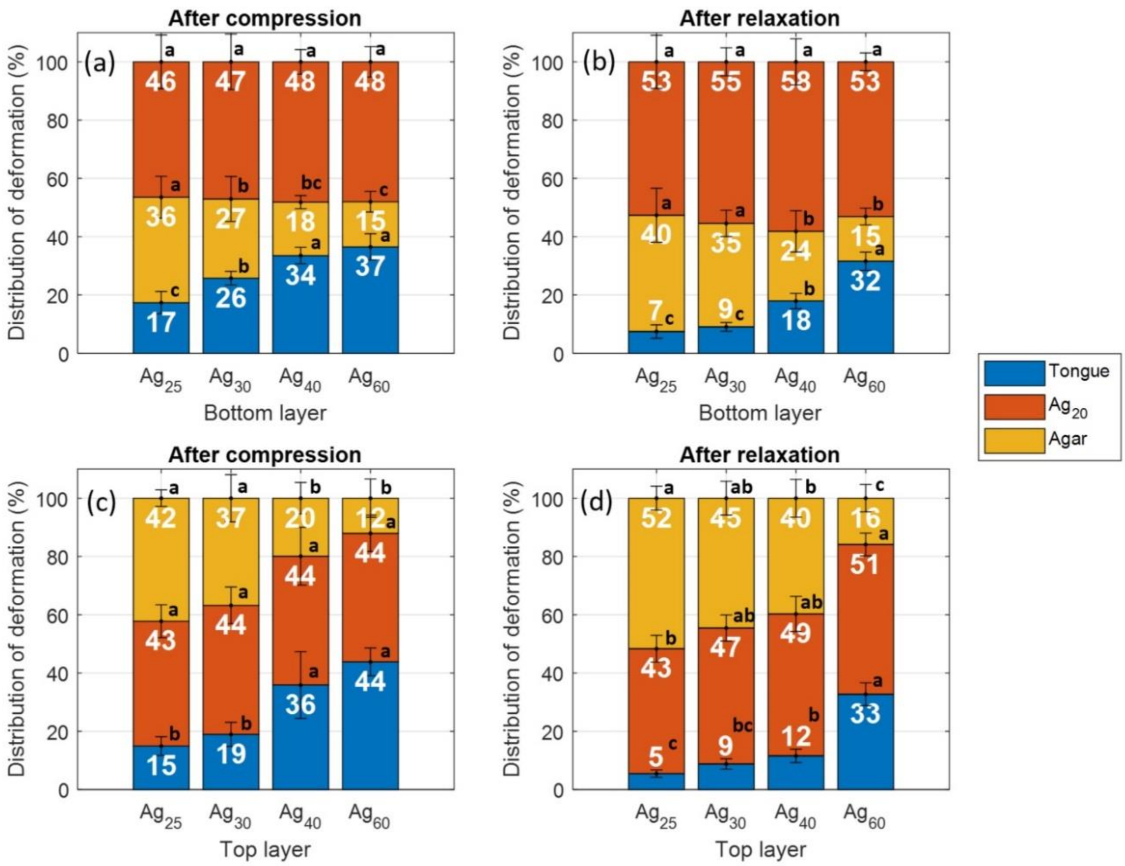



**Figure 4.5: Stacked bar graphs of the mean values and standard deviations of the relative deformation (%) undergone by the ATM ($\varepsilon_T$), lower gel layer ($\varepsilon_{L1}$), and upper gel layer ($\varepsilon_{L2}$), compared to total system deformation. Depicted here are the values for stacked gels in which an $Ag_{20}$ layer was paired with another agar layer ($Ag_{25}$, $Ag_{30}$, $Ag_{40}$, or $Ag_{60}$). $Ag_{20}$ was on top (layer $L_2$) in (a) and (b) and on bottom (layer $L_1$) in (c) and (d). Deformation at the end of compression step ($t_1$) is depicted in (a) and (c), and deformation at the end of holding step ($t_2$) is depicted in (b) and (d).**

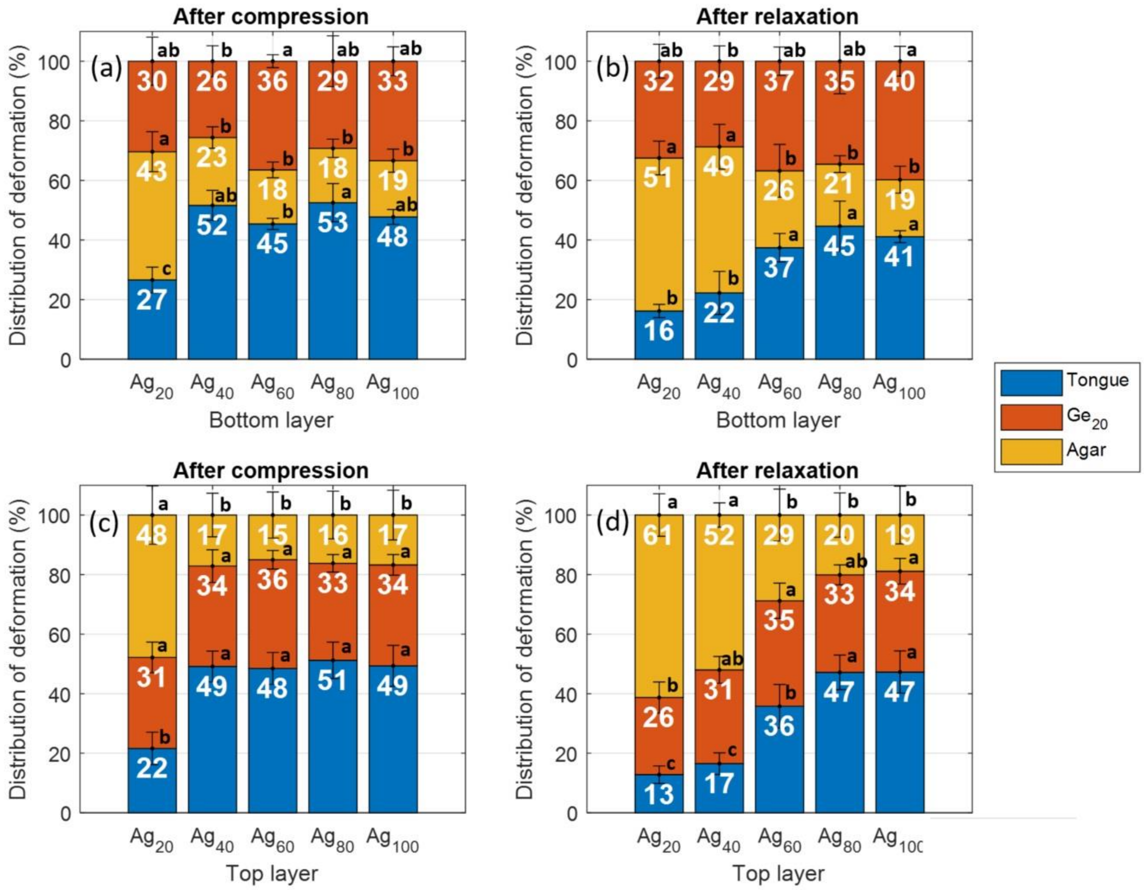



**Figure 4.6:** Stacked bar graphs of the mean values and standard deviations of the relative deformation (%) undergone by the ATM ($\varepsilon_T$), lower gel layer ($\varepsilon_{L1}$), and upper gel layer ($\varepsilon_{L2}$), compared to total system deformation. Depicted here are the values for stacked gels in which a Ge20 layer was paired with an agar layer (Ag20, Ag40, Ag60, Ag80, or Ag100). Ge20 was on top (layer $L_2$) in (a) and (b) and on bottom (layer $L_1$) in (c) and (d). Deformation at the end of compression step ($t_1$) is depicted in (a) and (c), and deformation at the end of holding step ($t_2$) is depicted in (b) and (d).

## 4.4. Conclusions

The ultrasound method described here was used to investigate how polymer type (agar or gelatin) affected the mechanical responses of stacked gels uniaxially compressed upon a biomimetic tongue. More specifically, the method made it possible to quantify the relative deformations of the individual components of the whole system: the artificial tongue model and the two gels layers. The degree of deformation was affected by polymer type (gelatin vs. agar), gel mechanical properties (degree of elasticity vs. brittleness), the specific combination of the two layers, and layer position (bottom vs. top). Changes in the gelatin concentration of the layers had no impact on their mechanical responses, while the opposite was true in the case of changes in agar concentration. This result most probably stems from the fact that gelatin is more elastic, while agar is more brittle.

In a real food oral processing context, the contribution of the ATM to the mechanical breakdown of this type of food is probably minor when compared to mastication. In this study, the compression of the gels by a flat probe (mimicking palate) also led to fracture of only a minority of gel types. The results, however, show that manipulation between the tongue and the palate can reveal a wide range of mechanical properties through the gels and may still greatly allow the texture to be assessed.

In summary, ultrasound methods show great promise for research seeking to investigate the mechanical behaviors of heterogeneous foods in contact with the tongue during oral processing. Beyond providing information about changes in the tongue's mechanical status over time, the methodology described here could be used to explore the mechanical phenomena that occur inside foods, which is impossible using traditional texture assessment techniques. Our future work will focus on more complex foods with more local heterogeneities. Multi-element ultrasound probes and 2-D imaging could also help to trace volume changes in such artificial tongue models and foods, and to take us further towards monitoring more complex heterogeneities, as well as for detecting and localizing cracks that occur within the gels during fracture.

## Acknowledgments

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# V
# Towards a new biomimetic setup

# Preface

*The work presented in the two previous chapters has demonstrated the interest of ultrasonic methods to describe the mechanical behavior of the tongue-food-palate system during compression. In particular, the biomimetic characteristics of the artificial tongue model used (deformable and rough) proved to be very important, with an impact on the physical phenomena both at the level of the interfaces and the volumes of the different components. However, as we know, food oral processing is much more complex and uniaxial compressions are only representative of small part of the very complex sequences of movements that govern the manipulation of food between the tongue and the palate. In particular, it is known that the shearing motions between the tongue and palate play an important role in texture perception mechanisms. To integrate more complex motions and continue the development of original instrumental methods, a new setup was required, which takes benefits from the knowledge and challenges encountered from our first setup.*

*The first part of this chapter presents the different stages of the design of this new biomimetic system, as well as the different instrumental methods it includes. In the second part of this chapter, the feasibility of the set-up was tested by measuring how friction coefficient values were affected by food properties (viscosity, particle presence), tongue properties (surface roughness, bulk rigidity), and operational parameters (normal stress, shearing velocity). This chapter is presented in the form in which it was published in the peer-reviewed journal - Food Hydrocolloids.*

# V (A)
# Setup development

## 5a.1. Requirements of the system

The design of this new prototype began with the development of a set of specifications. First of all, we decided to take advantage of the experience gained in previous work by considering rough and deformable artificial tongues with the PVA model. As for the palate, for the sake of simplicity, we wanted to continue the work with a rigid and smooth material.

Secondly, the aim was to be able to subject the food to compression and shear movements. We therefore looked for translational elements that could handle both types of movement, with properties relevant to the manipulation between the tongue and the palate (amplitude of movement, speed ranges), while guaranteeing a high level of precision in their control (fineness of movement, programming of customized sequences).

Thirdly, the system had to be adapted to embed a number of sensors, in order to propose several instrumental methods to characterize the impact of the displacement sequences on the mechanical properties of the system. Among them, it was necessary to consider a force sensor, as is done on the texture analyzer. We chose a three-axis sensor, in order to be able to characterize the three-dimensional forces applied to the palate. Then, we obviously tried to implement the ultrasonic sensors used in the previous studies, in order to continue their development.

Finally, all of the prototype's electronic components (motor elements and measurement elements) had to be controlled from the same user-machine interface in order to (i) control the movement of the various translation plates (during the set-up, calibration, measurement and shut-down phases) and (ii) ensure the acquisition of data from the various sensors in real time to allow their subsequent analysis.

## 5a.2. Setting up the device

Once the specifications were established, the design work of the new prototype was articulated around two main parts in parallel: a "Hardware" part dedicated to the design and the mechanical assembly of all the hardware elements of the prototype, then a "Software" part dedicated to the development of a user interface for system control, visualization and acquisition of data.

### 5a.2.1. Mechanical design

A schematic diagram and a picture of the device are presented in figure 5a.1 and 5a.2, they allow an overview of the experimental system. There were elements like translations stages, sensors (force, accelerometer and ultrsound) which were comercially available and there were also parts like the tongue holder and artificial palate that had to be designed inhouse. The different elements of this setup are discussed in details below.

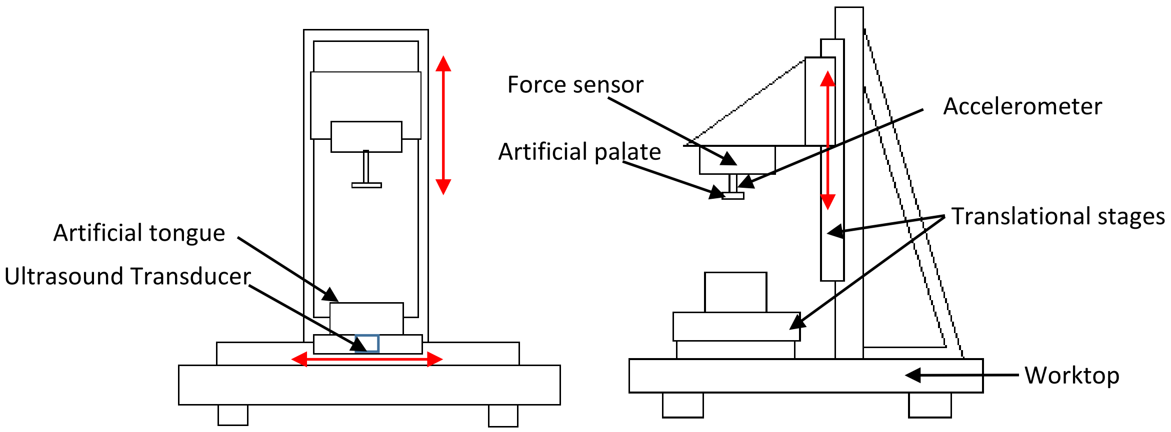



**Figure 5a.1: Schematic representation of the setup.**

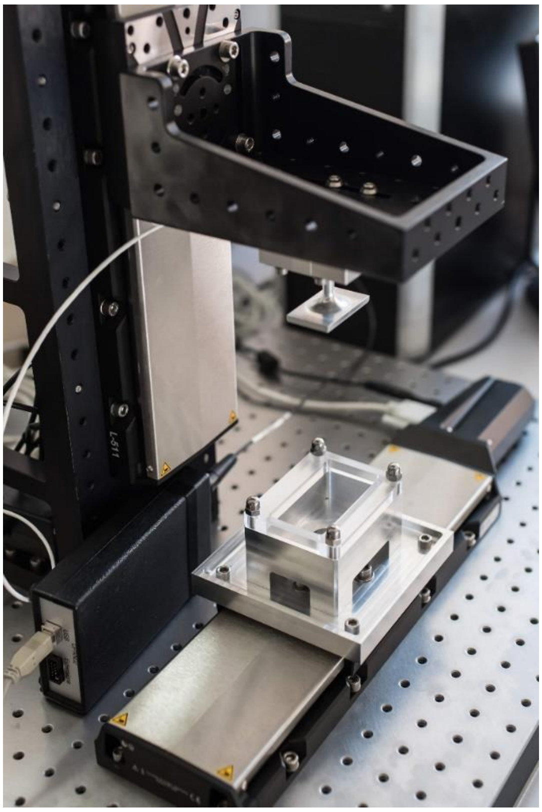
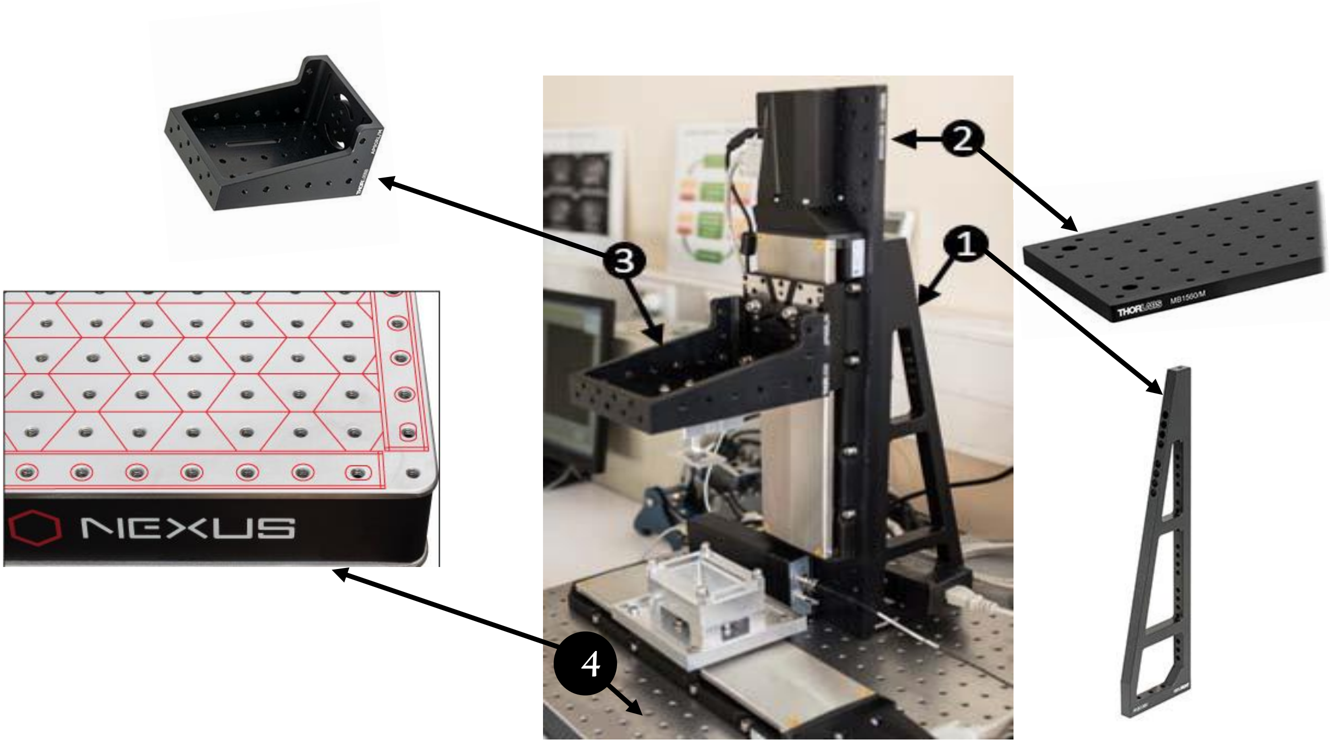


**Figure 5a.2: Photograph of the setup.**

<u>Structural elements</u>

Taking the scheme of the required setup as base, different parts constituting the structure of the prototype were ordered. To begin with the worktop, the entire system was mounted on a work surface (B4560L, Thorlabs, New Jersey, USA) designed to isolate vibrations through its honeycomb structure (Figure 5a.3). This work surface is supported by 4 sorbothane feet (AV6/M, Thorlabs). This allows further prevention of the parasitic vibrations due to the external environment.

On this work top, a horizontal translation stage could be fixed, on which the artificial tongue holder was attached. This translation stage was in charge of imposing shearing motions. Compressional motions were managed by a vertical translation stage for which support brackets




**<u>Figure 5a.3:</u> Structural elements of the setup: (1) bracket VB01B/M, (2) plate MB1560/M, (3) bracket AP90RL/M and (4) work surface B4560L.**

were needed (Figure 5a.3). One bracket (VB01B/M, Thorlabs) along with an aluminum plate MB1560/M, Thorlabs) was used give the attachment platform and support for the vertical translation stage. Another bracket (AP90RL/M, Thorlabs) was used to support the artificial palate (Figure 5a.3). All these elements together formed the skeleton of support for this new setup.

### 5a.2.2. Electronic elements

The different electronic elements of the setup were either the devices used to generate or control the compression and shearing movements, or the measurement systems that we wanted to implement to follow these movements. They are described one by one in this section.

Translation stages: In order to allow more complex movements, including both compression and shear between the tongue and the palate, the new device includes two translation stages (L-511.60AD10, PI, Germany) (Figure 5a.4) positioned perpendicularly.

**Figure 5a.4: Translation stage (L-511.60AD10, PI System).**

These stages have a range of motion of 155 mm with an accuracy of 0.05 µm and a minimum relative displacement of 0.488 µm. They allow precise movements to be performed at a maximum speed of 90 $mm.s^{-1}$. As seen previously, when swallowing liquid food, an average velocity of 10.34 ± 2.1 $mm.s^{-1}$ could be observed, in a range from 2.1 $mm.s^{-1}$ to 32.43 $mm.s^{-1}$ (Peng et al., 2000). The selected translation stages thus allow the reproduction of movements close to *in vivo* conditions, as well as the acquisition of the position and velocity of the mobile platforms of each stage at any time.

Force sensor:

In order to monitor the force applied at the palate-food interface, a 3-axis force transducer (K3D60a ± 50N, ME Systeme, Hennigsdorf, Germany) (Figure 5a.5) with a measurement range of ±50N with an accuracy class of 1% was chosen. Coupled with a C series Strain/Bridge input module (NI-9237, National Instrument, Austin, TX, USA), a sampling rate of up to 50 kHz can be achieved for signal digitalization.

**Figure 5a.5: Force sensor (K3D60a, ME Systeme).**

As seen previously, when swallowing liquid food, the tongue applies a pressure on the center of the palate that can vary between 25 kPa and 290 kPa (Kieser et al., 2008). We thus ensured that the measurement range of the force transducer was consistent with the chosen dimensions of the palate (discussed later). A 3-axis force sensor will allow to measure the forces during compression and shear, and thus to deduce the friction coefficient between the tongue and the palate.

Accelerometer:

In order to quantify the vibrations generated during the interactions between the surfaces of the artificial tongue and the artificial palate, a piezoelectric accelerometer (352A24, PCB Piezotronics, Figure 5a.6) was placed on the device. It has a measurement range of ±490 m/s² with an accuracy of 0.002 m/s². Coupled with an acoustic and vibration input module (NI-9234, National Instruments, TX, USA), a sampling frequency of up to 51.2 kHz can be achieved.


**Figure 5a.6: Accelerometer (352A24, PCB Piezotronics).**

Yet human mechanoreceptors perceive vibrations at a frequency between 0 and 600Hz (Amaied et al., 2015), so it is possible to study them with this equipment. This type of sensor has indeed already been implemented for similar applications, related to tactile perceptions during finger sliding on rough surfaces of various natures.

Ultrasonic analysis system:

This is the system used on the first prototype. It includes a single-element piezoelectric transducer (V103RM, Olympus) (Figure 5a.7) which allows the transmission and reception of compressional ultrasound waves with a central frequency of 1 MHz. A piezoelectric element placed between two electrodes vibrates when subjected to a voltage. This vibration allows the generation of the ultrasonic wave, which will propagate, then be reflected by the various interfaces of the studied system. The reflected acoustic energy then causes the piezoelectric element to vibrate, allowing, via the measurement of the voltage across the electrodes, the recording of the reflected ultrasound signal.

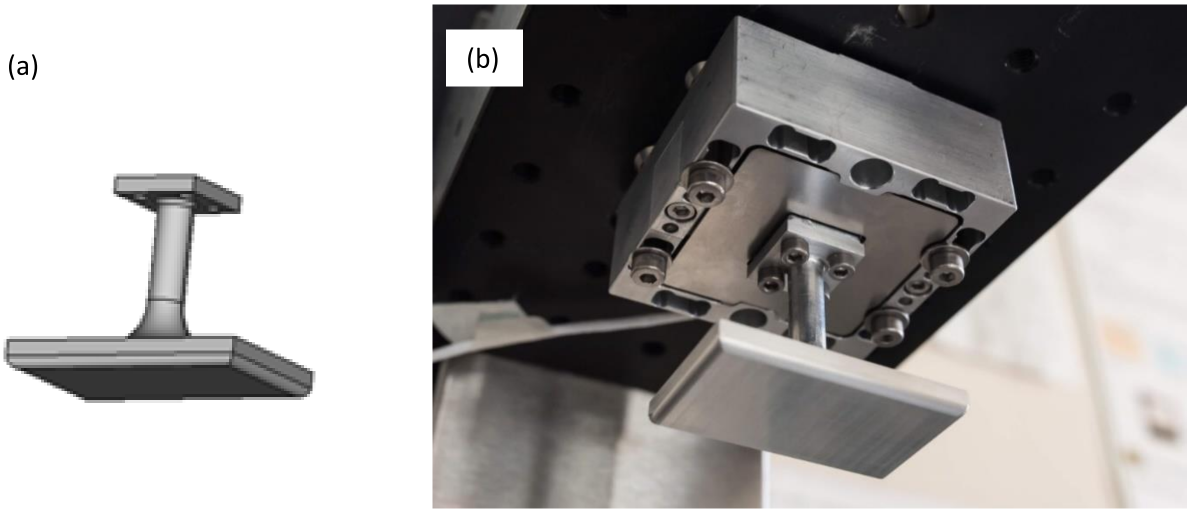
**Figure 5a.7: Ultrasound Transducer (V103RM, Olympus).**

The ultrasonic transducer was coupled to a generator-receiver allowing the emission of electrical signals and their digitization (Sonatronic). The box allows to control the different parameters of the negative square pulse used for the emission of the ultrasonic signal (voltage from 1 to 200 V, adjustable width from 30 ns to 1 µs), as well as the amplification of the received signal (0 to 40 dB). This system allows the digitization of signals on 12 bits and at 100 MHz, with a maximum frequency of recurrence of the signals (frequency of repetition of the ultrasound shots) of 90Hz.

### 5a.2.3. Development of custom-made parts

The implementation of shearing movements on the new prototype required a revision of the geometry of the artificial palate (circular shape on the first system). In the same way, the shape of the artificial PVA tongue had to be reworked to adapt to the new experimental setup. Several specific situations led to design custom mechanical parts. FreeCAD computer-aided design tool was used in order to precisely define the plans of the necessary parts. Once the parts were designed, they were machined by Volpi S.R.O., Brastislava, Slovakia.

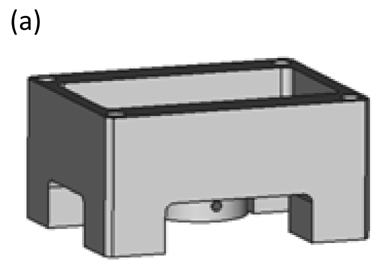
<u>Artificial palate:</u>




**<u>Figure 5a.8:</u> (a) 3-D view of the artificial palate by computer aided design (b) photograph of the palate attached on the setup.**

The artificial palate (3-D view of the CAD file provided in Figure 5a.8) was attached to the force sensor (itself attached to the bracket of the vertical moving translation stage, Figure 5a.8). An artificial palate of circular shape (as could be found on the previous device) was no longer relevant for the new system because of the addition of a shear axis displacements. We had to ensure that at all times during the experiments the ultrasound beam was facing the flat surface of the palate. Indeed, if this were not the case, we might encounter fluctuations in the shape of the ultrasound signals which would not only reflect mechanical phenomena at the interface between the tongue and the palate, but also geometric changes in the part of the palate which enters the field of the ultrasound beam. As with the uniaxial compression experiments in the previous chapters, the ultrasonic transducer was fixed in contact with the lower surface of the artificial tongue. In this way, the transducer moved with the tongue during the shearing movements. The shape and dimensions of the palate (rectangular, 45 mm x 25 mm) were thus defined according to the size of the ultrasound beam (around 20 mm), and the amplitude of the shear displacements that we wished to consider with the device (20 mm). This technical choice results in a large contact surface between the tongue and the palate. It makes sense in relation to the actual contact area between the tongue and the palate during the oral process. It also allows the study of the impact of heterogeneous foods, which is original in comparison with classical tribology approaches. In order to avoid damaging the tongue on the sharp edges of the palate, a slight fillet with a radius of curvature of 2 mm was made on all edges of the rectangular side of the palate that comes into contact with the artificial tongue.

Tongue support and artificial tongue models:



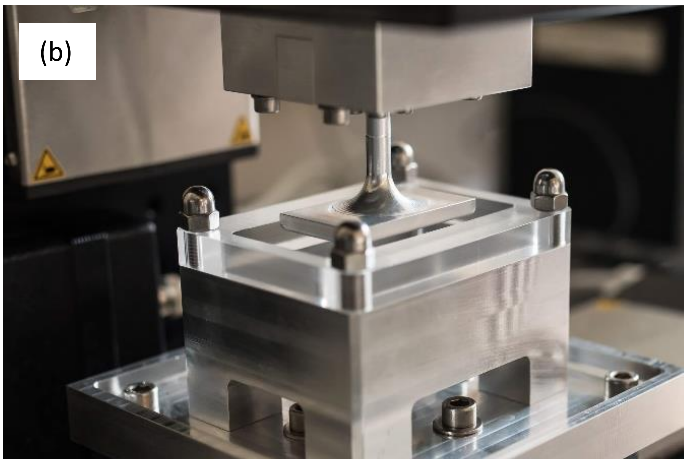
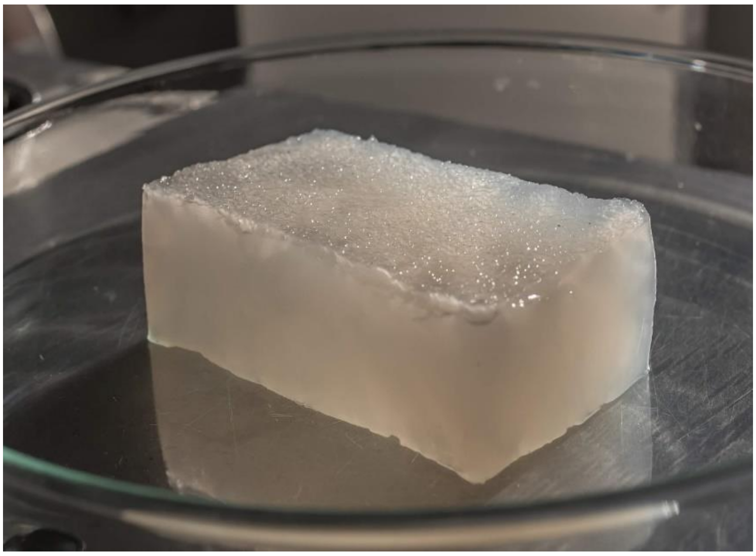




**Figure 5a.9: (a) 3-D view of the tongue holder by computer aided design (b) photograph of the tongue holder attached on the setup.**

On the horizontal stage, a system was required to be designed to serve as a support in order to position the artificial tongue sample on the translation stage carriage used to perform the shearing movements (Figure 5a.9). The tongue holder was also designed to be able to fix the ultrasonic sensor directly in contact with the lower surface of the artificial tongue. The dimensions of the artificial tongue (and of the cavity into which it is inserted) have also been adapted from the work presented in the previous chapters. It was necessary to ensure that at all times during an experiment, when the tongue holder moves to generate the shearing movements, the surface of the palate remains within the perimeter of the tongue surface. We therefore chose an artificial tongue with a parallelepiped shape, 80 mm long and 45 mm wide.

In a similar way to what has been done and described previously, we have chosen to work with artificial tongue models made in PVA (Figure 5a.10). New parallelepiped molds have been designed, always with one of the faces covered with abrasive paper (references P40 and P80). Two to five cycles of freezing (16 hours at -20°C) and thawing (8 hours at 20°C) were imposed in order to give it its structure and mechanical properties. The change in the dimensions of the artificial tongue models implied differences in the temperature kinetics during the freezing and thawing cycles. It is known that these kinetics can have a strong impact on the resulting mechanical properties. Thus, the elastic properties of the resulting PVA gels were measured by performing compression tests.



**<u>Figure 5a.10:</u> A PVA based parallelepiped artificial tongue model.**

### 5a.2.4. Design of a user-machine interface

Once all the components of the experimental system were in place, a user-machine interface was programmed in order to ensure the control of the system. This interface was developed with the LabVIEW software.

<u>Control of the movement of the plates</u>

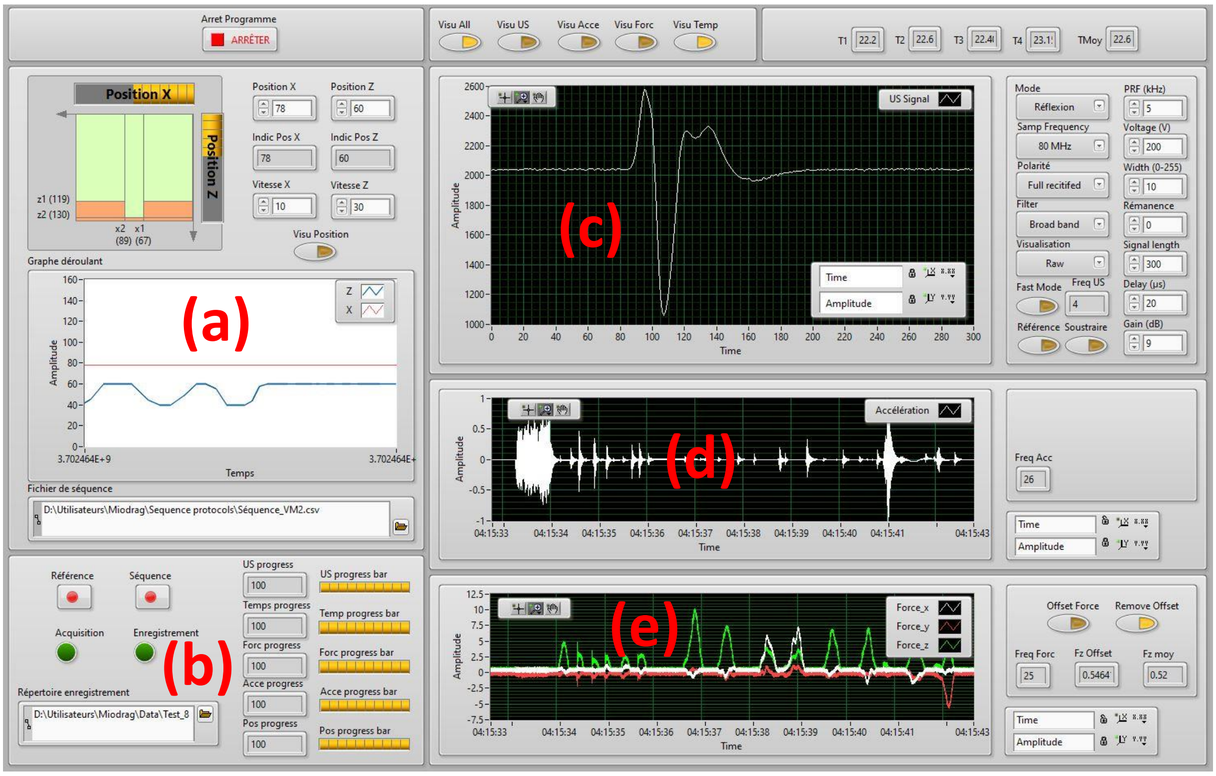
The program allows first of all the manual movement of the two translation plates (Figure 5a.11a). This allows, for example, to put the artificial palate in a high position while the artificial tongue and the food are put in place, and then to adjust the two plates until the desired initial position is reached to launch a test. Then, the program allows the reading of a sequence of displacements which is recorded in a spreadsheet (Figure 5a.11b). It reads, line after line, the displacement orders (position, speed) or pause times corresponding to the sequence that we wish to execute.

Visualization and acquisition of instrumental measurements

The second part of the program allows the visualization in real time of the various measurements made possible by the device. The temperature values from four thermocouples are thus reported. In our case, the experiments were carried out at a constant temperature (20°C) provided by the air conditioning of the laboratory. It is thus a simple measurement to ensure the stability of the temperature.

Then, a first graph with the ultrasonic signal as it was measured previously is shown (Figure 5a.11c). The interface allows to control the properties for the emission of the signal (voltage, width), as well as the characteristics for its digitization (gain, window size).

Another graph allows to report the measurements from the accelerometer (Figure 5a.11d), while the measurements from the three-axis force sensor are visualized in the last graph (Figure 5a.11e). The program allows the recording of data from the different sensors (position, temperature, ultrasound, acceleration and force) during two stages. The first one corresponds




**<u>Figure 5a.11:</u> An overview of the Labview user-interface, (a) displacement amplitude of the translation stages, (b) data acquisition window, (c) ultrasound monitoring window, (d) accelerometer monitoring window and (e) force monitoring window.**

to a so-called reference phase, when the palate and the tongue are not in contact. This allows an accurate quantification for a period of two seconds of the measurements in the absence of loading. The second period of course corresponds to the duration of the sequence programmed using the spreadsheet. All the raw data are then imported on the Matlab software to allow their visualization, and their processing.

## 5a.3. Proof of concept study

The device thus conceived fulfills all the constraints of the specifications that we had set ourselves. It offers many possibilities to simulate complex movement sequences mimicking the manipulation of food between the tongue and the palate. Moreover, it offers a large number of possibilities for instrumental measurements in order to characterize the impact of these sequences of movements on the mechanical properties of the tongue-food-palate system.

Faced with this vast range of opportunities, we had to make choices, while continuing the approach which was ours to gradually increase the complexity of the phenomena. It thus appeared important to us to concentrate on the first novelty brought by this system: the possibility of considering shearing movements in the presence of food between the tongue and the palate. Since the device is quite new, we have chosen to consider simple sequences of movements under constant vertical position of the palate, where cycles of back and forth movements in shear (at constant speed) and spaced by pause times were imposed, like what can be done in classical tribological analysis protocols.

Under these conditions, the palate is supposed to remain at a constant distance throughout the tests. The ultrasonic response is therefore not assumed to vary in terms of time of flight, except if friction between the tongue and the palate induces significant vibrations in the vertical direction. However, this type of vibration cannot be observed with the ultrasonic system considered here, as the frequency of the ultrasonic shots is too low (about 90Hz). Thus, the observation of the M-Mode image of the ultrasonic signals during a test sequence (see Figure 5a.12) allows us to observe slight fluctuations in the time of flight related to parallelism defects between the surface of the palate and the axis of movement of the horizontal translation plate (although minor, evaluated at less than 0.5°).

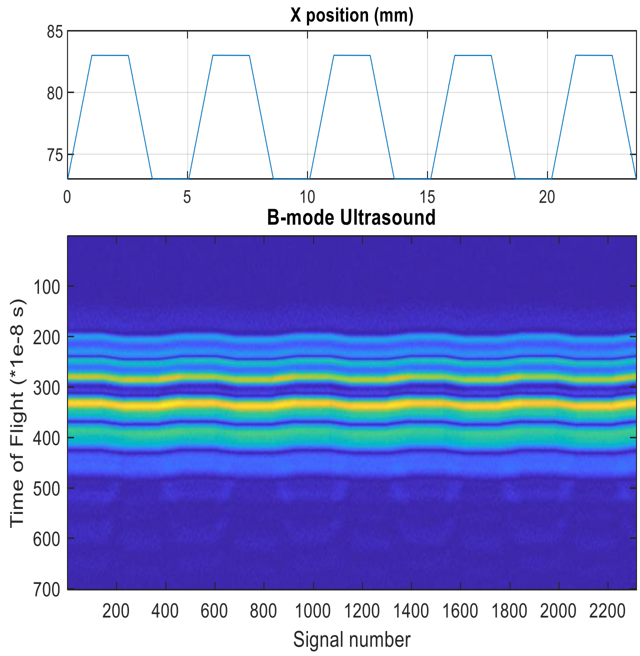



**Figure 5a.12: Variation of ultrasound ToF with respect to the displacement of the horizontal stage.**

The vibrations associated with the friction between the tongue and the palate are more easily quantified with the accelerometer. For example, it is clearly seen (Figure 5a.13) that the phases of displacement of the horizontal plate coincide well with significant vibratory events. However, it is necessary to take into account the fact that the translation plates also induce vibrations and that the vibration at the tongue-feed-pallet interface is therefore probably not the only component recorded. The analysis of the vibration signal is therefore complex. The development of each of these methods requires a significant investment that we considered too ambitious for this single thesis work.

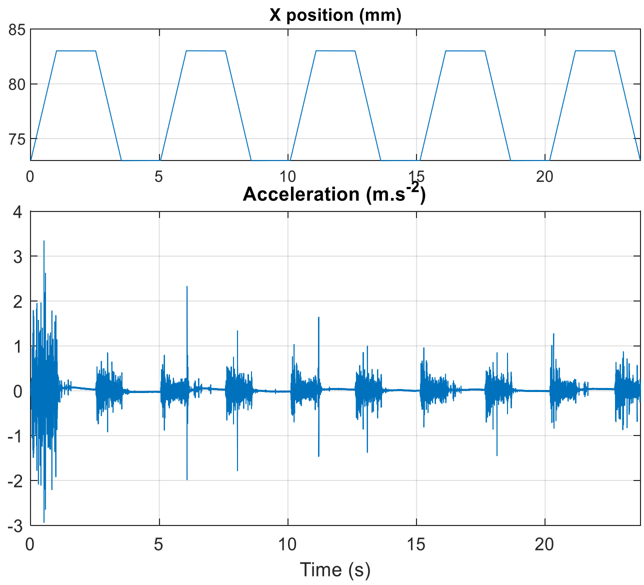



**Figure 5a.13: Variation of acceleration with respect to the displacement of the horizontal stage.**

We have therefore turned to the measurement of friction forces allowed by the three-axis sensor (Figure 5a.14). Not because it was simpler (it may not be) but because the measurements associated with this sensor can be related to tribology analyses for which has gained a lot of attention in recent years by the food oral processing community. This allows us to have a certain number of reference points for the validation of the proof of concept of our newly designed biomimetic device. This is the purpose of the work described in the section (Chapter 5B).

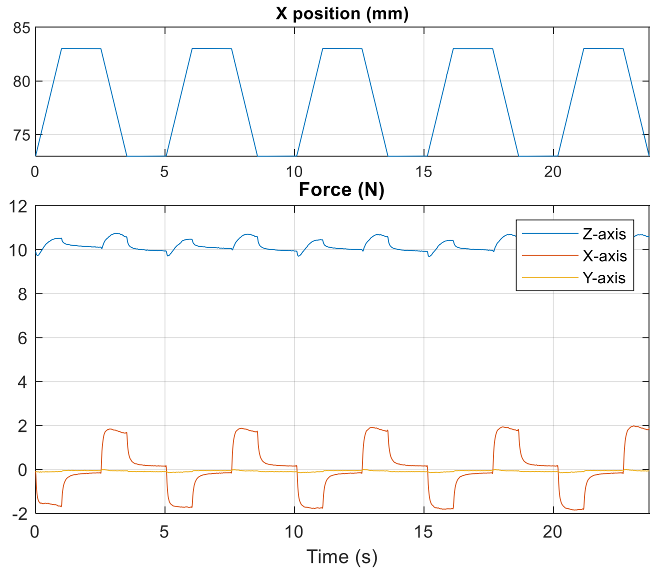
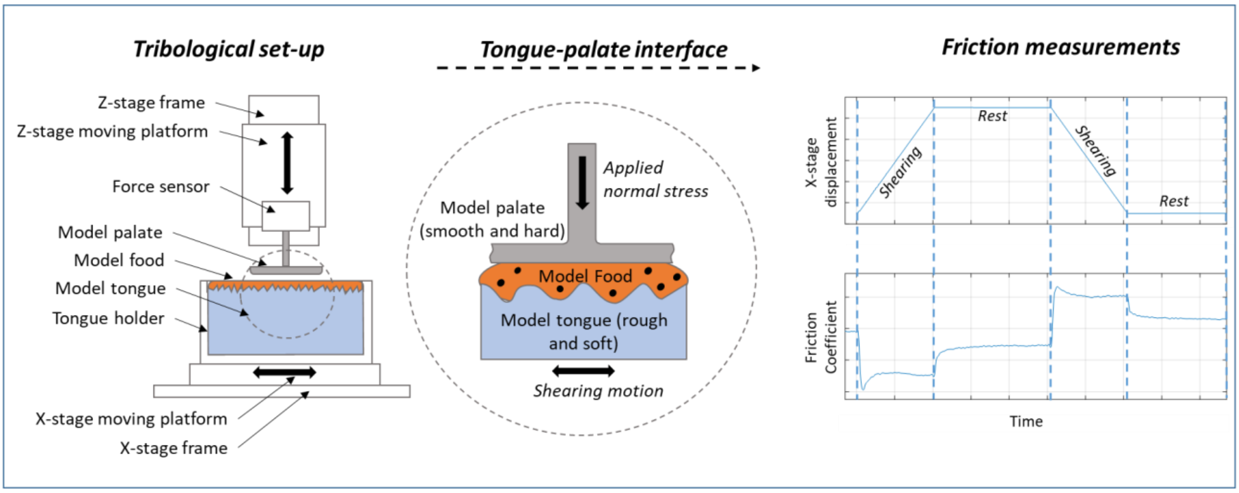



**Figure 5a.14: Variation of force with respect to the displacement of the horizontal stage.**

# V (B)

# A new biomimetic set-up to understand the role of the kinematic, mechanical, and surface characteristics of the tongue in food oral tribological studies

# Graphical abstract



## 5b.1. Introduction

The quest to understand and model consumers' sensorial perceptions of food is now of paramount interest in efforts to develop products for target populations. Food texture perception is an important aspect of the overall sensorial appreciation of food. This physiological process has recently gained the attention of the scientific community, and especially that of researchers seeking to elucidate the mechanisms behind perceptions like mouth feel and astringency. Soft tribology has emerged as a prime technique for investigating friction phenomena between soft oral surfaces and food during oral processing. There is a growing body of research dedicated to these oral tribological phenomena that describes the dependence of friction forces on lubrication and surface properties and the resulting impact on different sensorial perceptions pertaining to texture and mouth feel (Krop, Hetherington, Holmes, Miquel, & Sarkar, 2019; Liu, Stieger, van der Linden, & van de Velde, 2015; Stokes, Boehm, & Baier, 2013). In a recent review, Panouillé, Saint-Eve, & Souchon (2016) summarized the instrumental and sensorial methods regularly employed to understand food perceptions. The authors underscored that linking physical measurements (mechanical, rheological, and tribological) to sensorial assessment methodologies can be difficult, mainly due to challenge of replicating in vivo conditions. Rudge, Scholten, & Dijksman (2019) have further detailed the challenges tied to relating tribological measurements and the perceived textural attributes of food that arise from the complexity of the oral cavity (contact mechanisms, motions, surface roughness, rigidity, and lubrication).

Linking tribological results with sensory perceptions is also arduous because of the adaptability constraints of commercially available instruments and the inability of contact mechanisms and surface properties to better mimic real oral physiological conditions. The limited selection of contact geometry, (e.g., ball-on-disc, pin-on-disc), movements (linear or rotating), and surfaces is highly restricting. It is also important to point out that, in most of the oral tribology studies where a small contact area is applied, the suspended heterogeneities (closer to the threshold of particle perception) in semisolid or aqueous foods are often ignored. These constraints have been highlighted by Pradal & Stokes (2016), who rightly argue that there is a need to choose a "physiologically relevant tribological system" in order to apply tribology as a measure for predicting texture perception. Many recent studies have therefore tried employing custom-built tribological set-ups (Chen, Liu, & Prakash, 2014; Mo, Chen, & Wang, 2019), which are mainly designed to allow for greater flexibility in adapting motion, normal stress, shearing velocity, and contact geometry as needed. Therefore, in-house development or customization of tribometers to mimic in-mouth movements could be a solution (Rudge et al., 2019).

Furthermore, when looking for suitable oral surface mimics, it should be kept in mind that the human tongue is a complex muscular organ with a peculiar surface topology. Its dorsal surface bears several types of papillae that play a major role in mechanical interactions with food. There are four types of papillae on the surface of the tongue: filiform, fungiform, folate, and circumvallate (Jung, Akita, & Kim, 2004; Ranc, Servais, Chauvy, Debaud, & Mischler, 2006). The filiform papillae are present at high densities across the tongue's surface, greatly contributing to its overall roughness. The human tongue is also a complex biological tissue from a mechanical perspective. Napadow, Chen, Wedeen, & Gilbert (1999) have described the tongue as a hydrostatic organ that keeps its volume constant during compression or motion but that simultaneously changes its rigidity. The shear elastic modulus of the human tongue has been measured using several techniques: mechanical indentation (G = 0.38 kPa) (Gerard, Ohayon, Luboz, Perrier, & Payan, 2005), mechanical compression (Ishihara et al., 2013), and magnetic resonance elastography (G = 2.67 kPa) (Cheng, Gandevia, Green, Sinkus, & Bilston, 2011). The tongue's stiffness, however, changes significantly between its "rest" state (E = 12.2 ± 4.2 kPa) and its "contracted" state (E = 122.5 ± 58.5 kPa) (Ishihara et al., 2013).

Peculiarities of the tongue tissue such as rigidity and surface roughness might have a complex and synergistic role in oral tribology; they must therefore be well accounted for in experimental set-ups. Attempts have been made to recreate real physiological parameters as best as possible by using biological tongue tissues or polymer surfaces that can mimic the wettability and deformability of the tongue (Carpenter et al., 2019; Dresselhuis, de Hoog, Cohen Stuart, & van Aken, 2008). However, there are some constraints associated with these novel surfaces as well. Once biopsied, biological tissues rapidly lose their mechanical properties, making it difficult to preserve the tissue's surface profile and rigidity. Artificial surfaces, such as those made with polydimethylsiloxane (PDMS), need to be carefully modified to properly replicate surface roughness, rigidity, and wettability.

In this study, we present a custom-built tribological set-up with the aim of better integrating the surface, mechanical, and kinematic characteristics of the tongue. The greater objective is to create a set-up that can enhance our understanding of the changes in friction forces over time during the oral processing of food. The set-up was designed to have a large degree of contact in tongue-palate geometry, thus resembling real oral anatomy, and to provide flexibility in terms of shearing velocity and displacement. Also, a novel tongue-mimicking sample (TMS hereafter) made of a polyvinyl alcohol (PVA) hydrogel was used. PVA is a water-soluble synthetic polymer that is well known for its application in biomedical engineering, namely for developing artificial soft tissues and mimicking oral mucosa (Chatelin et al., 2014; Fromageau et al., 2007; Gennisson et al., 2007; Jiang, Liu, & Feng, 2011; Mamada, Fridrici, Kosukegawa, Kapsa, & Ohta, 2011). The rigidity of the hydrogels made from PVA can also be controlled by varying the number and the kinetics of freezing and thawing cycles. This ability to customize the rigidity and imprintability of the surface roughness profiles of hydrogels made PVA a suitable fit for our study. To investigate the feasibility of this new set-up, cottage cheese with or without suspended cellulose particles was used as a model food. It allowed the explicit incorporation of microscale heterogeneities into the food system and an analysis of how friction was affected by normal stress, shearing velocity, TMS roughness, and TMS rigidity.

## 5b.2. Materials and methods

### 5b.2.1. Model food system

Cottage cheese (Calin extra [0% fat], Yoplait, Boulogne-Billancourt, France) with and without suspended cellulose particles (5% w/w; Vivapur, JRS Pharmaceuticals, Rosenberg, Germany) was used as a model food. The median sizes of the two sets of cellulose particles used in this study were 15 µm (Vivapur 105) and 250 µm (Vivapur 200); in this article, they are hereafter referred to as "small" and "large" particles, respectively. The choice of non-fat cottage cheese as the carrier medium stemmed from the fact that, in one of our preliminary sensory studies, panelists were able to well rank the levels of added heterogeneities in this particular medium (Tournier, Poette, Rachidi, Septier, Martin, & Feron, 2018). Furthermore, the cottage cheese was used in both "undiluted" and "diluted" forms. The diluted cottage cheese was mixed with water (50% w/w). The 50% dilution factor was chosen to mimic the dilution of cottage cheese with saliva when the food is in close proximity to the tongue surface (Brodkorb et al., 2019; Doyennette et al., 2014; Laguna, Farrell, Bryant, Morina, & Sarkar, 2017). Solutions of glucose syrup (Caullet, Erquinghem-Lys, France) made with water were used as Newtonian fluids for rheological and tribological comparisons; three different concentrations (0%, 49%, and 67% w/w) were employed.

A strict protocol was developed after a series of preliminary tests to ensure that the different model food types had experienced the same mechanical history before being used in the experiments (the tribological and rheological tests). The different model food types were thus prepared in identical quantities (8 g) and in identical containers. Once all the ingredients had been added, the mixtures were subject to magnetic stirring (750 rpm, 20°C) for a period of 30 minutes, just before being used in the different experiments. However, it is important to mention that the stirring did not cause any rheological changes to the system, as confirmed by measuring the viscosity of samples (protocol described below) throughout the course of a complete set of experiments.

For the viscosity measurements of the cottage cheese types, a rheometer (HAAKE RheoStress 600, Thermo Fisher Scientific, Waltham, USA) with parallel-plate geometry (d = 35 mm) was used to obtain flow curves (at 20 °C, with shear rates between 2 and 450 $s^{-1}$ and a 1 mm operating gap); five replicates were performed. The absence of time dependency of the foods' rheological properties was also validated by checking the agreement between the flow curves obtained for increasing and decreasing shear rates.

The viscosity values of the water and the glucose solutions were measured using cone-plate geometry (d = 60 mm, α = 2) at 20 °C. The applied shear rate was between 2 to 450 $s^{-1}$, and an operating gap of 90 μm was employed. Due to sensitivity issues, especially at the lower shear rate, only the mean values obtained over 10 $s^{-1}$ were plotted, which assumed that the viscosity of the Newtonian fluids remained independent of the shear rate. The measured mean values were 1.2 mPa.s ± 0.1, 10.9 mPa.s ± 0.4, and 52.9 ± 0.4 mPa.s for the water, the 49% glucose solution, and the 67% glucose solution, respectively. These values are also in accordance with those found in our previous study (Mathieu et al., 2018).

### 5b.2.2. Development of the bio-mimicking tongue-palate set-up

The development of the bio-mimicking oral tribological set-up aimed to simulate more realistic contact geometry, surface pairing, and shearing motion. The process thus required carefully selecting and assembling commercially available structural parts as well as designing and fabricating tailored parts (Figure 5b.1). These steps are discussed in detail below.

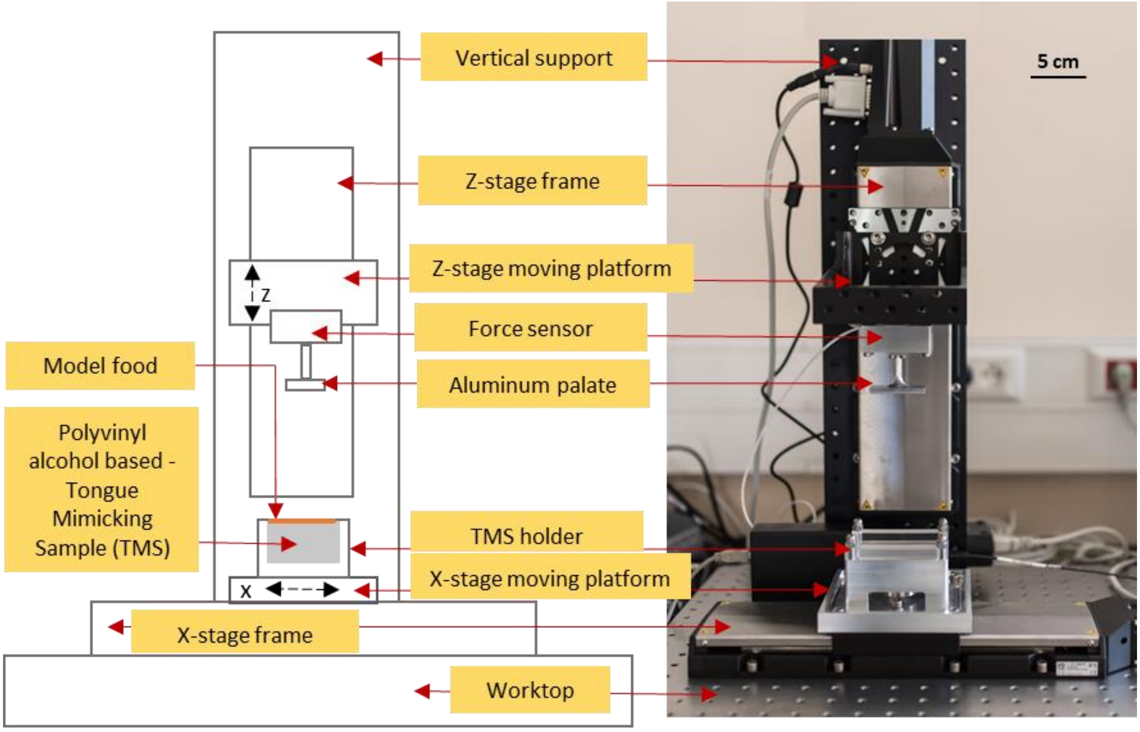



**Figure 5b.1: Schematic representation (left) and photograph (right) of the experimental set-up. At the top of the set-up is a rectangular aluminum plate (acting as the palate) that is mounted on a 3-axis force sensor. This sensor is attached to the z-axis moving platform, which is used to apply a controlled normal load. At the bottom of the set-up, a film of liquid food is deposited and spread across the surface of a tongue-mimicking sample (TMS). The position of the TMS is secured by a holder attached to the x-axis moving platform, which creates the shearing motion between the TMS and the aluminum palate.**

### Fabrication of the tongue-mimicking samples

The TMSs (Figure 5b.2a) employed in this study were made from PVA solutions. To begin with, PVA (MW 89,000 - 98,000, 99% hydrolyzed (degree of saponification), Sigma Aldrich, Saint-Louis, USA) was dissolved in ultra-pure water (10% w/w) and then subject to constant magnetic stirring at 80 °C for 2 h. The solution was subsequently cooled to room temperature. Cuboidal molds (80 mm x 45 mm, 25 mm in height) were completely filled with the solution.

To produce rough surfaces, the bottom rectangular side of the molds was covered with a sheet of P40 (grain size = 425µm, ISO/Federation of European Producers of Abrasives) sand paper (Norton, Saint-Gobain, France). This approach was taken to simplify the surface topology for this first feasibility study. The real surface of the human tongue has a complex topography, with asperities whose heights range from 42.5 to 101.4 µm (Uemori, Kakinoki, Karaki, & Kakigawa, 2012); this complexity is difficult to replicate in vitro. In this study, two contrasting surface profiles were prepared: one without sand paper (referred to hereafter as “smooth”) and the other one with P40 sand paper (referred to hereafter as "rough"). The molds were then sealed and subjected to cycles of freezing and thawing, which helped to achieve the desired level of rigidity (Fromageau et al., 2007). During each cycle, the TMSs were frozen at -20 °C for 16 h and then thawed at 18°C for 8 h. Finally, the TMSs were unmolded and stored in reverse-osmosis-treated water at room temperature (20°C) for several months. The Young's moduli of the TMSs were estimated by performing uniaxial compression tests using a texture analyzer (TA.XT plus, Stable Micro System, Surrey, United Kingdom; compression speed: 1 mm/s; strain rate of up to 20%; at least 3 replicates performed). Two types of TMSs were obtained—soft TMSs after 3 cycles (referred to hereafter as "soft"; 50.17 ± 1.46 kPa), and hard TMSs after 5 cycles (referred to hereafter as "hard"; 100.78 ± 2.12 kPa).

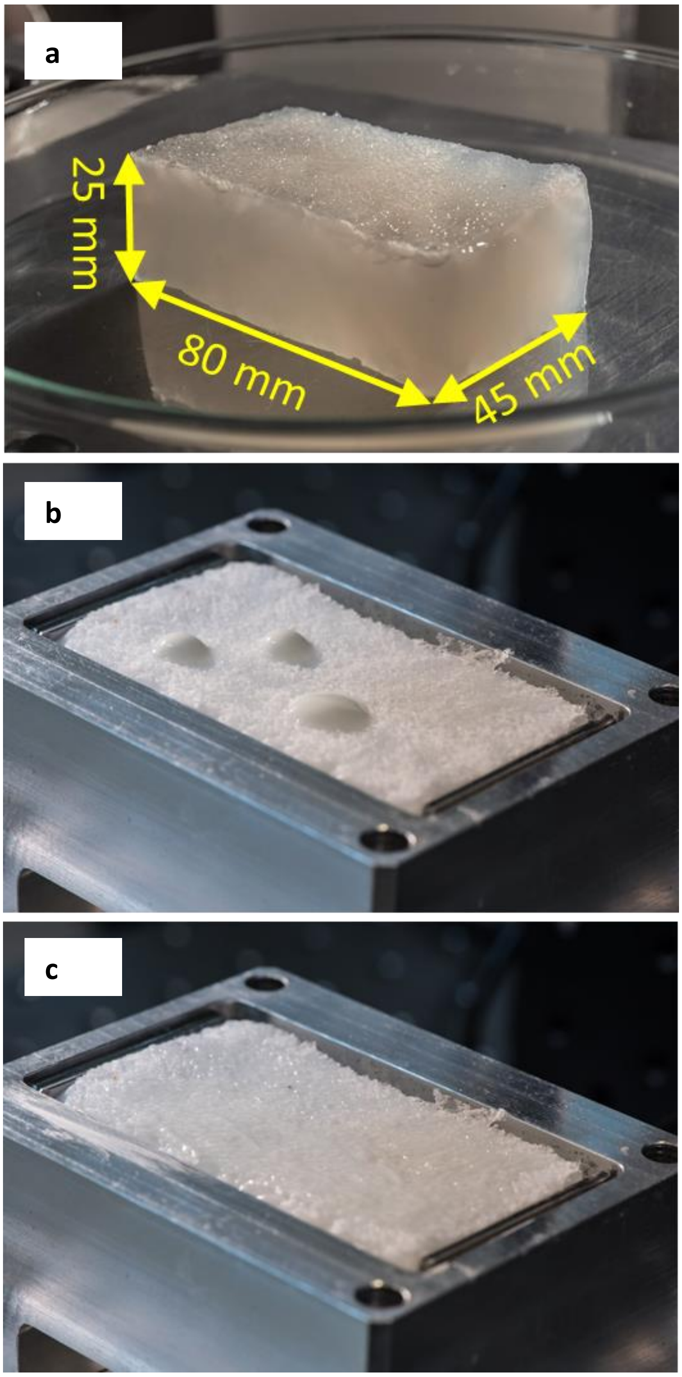



**Figure 5b.2: (a) Parallelepiped tongue-mimicking sample (TMS) made with polyvinyl alcohol; (b) the TMS in the device's holder, covered by 0.25 g of cottage cheese (in the form of three droplets); and (c) cottage cheese homogeneously spread across the whole surface of the TMS with a spatula right before positioning the TMS in contact with the aluminum palate and launching a test. The visual aspect of the surface after a test was similar.**

Structural elements of the set-up

The device included two linear translation stages (L-511.60AD10, Physik Instrumente (PI), Karlsruhe, Germany) (Figure 5b.1) that were positioned perpendicularly over a worktop (B4560L, Thorlabs, New Jersey, USA) designed to isolate the set-up from external vibrations. The translational stages had a displacement amplitude of 155 mm with an accuracy of 0.05 µm and a minimum relative displacement of 0.488 µm. The maximum displacement speed was 90 $mm.s^{-1}$. According to the literature, during the oral processing of liquid foods, an average tongue movement speed ranges from 2.1 $mm.s^{-1}$ to 32.43 $mm.s^{-1}$ (average 10.34 ± 4.92 $mm.s^{-1}$) (Peng & Miethke, 2000). The chosen translation stages made it possible to reproduce such in vivo movements. The set-up also comprised different tailored parts (Volpi S.R.O., Brastislava, Slovakia), including an aluminum plate (rectangular, 45 mm x 25 mm) that was attached to the vertical translational stage and that played the role of the hard palate. This aluminum surface was smooth relative to the surfaces of the TMSs. To control the force applied by the palate, a three-axis force sensor (K3D60a ± 50 N, ME Systeme, Hennigsdorf, Germany) with a measurement range of ± 50 N and an accuracy class of 1% was chosen. The horizontal translation stage on the other side was equipped with a rectangular holder for the model tongue.

Machine-user interface

A LabVIEW (National Instruments, Texas, USA) based interface was also developed to operate the biomimetic set-up. The interface was used to adjust the positioning of the translational stages and to impose specific sequences of motion by programing the stage motor controller (C.884.4DC, 4 channels, Physik Instrumente, Karlsruhe, Germany). The controller supported functions such as linear vector motion, point-to-point motion, and user-definable trajectories. It also served to record operating data related to stage position. In addition, to facilitate the acquisition of force, a bridge module (NI-9237, National Instruments, Texas, USA) was used to provide an efficient sampling rate (50 kS/s/ch).

### 5b.2.3. Test protocol

In the first step of the test protocol, a TMS was taken out of the water in which it had been stored; excess surface water was gently removed by absorbent paper until no traces of water were seen on the paper. The TMS was then placed into the cavity on the horizontal translation stage. The experiments were performed at room temperature (20° C) because it was hard to match the surface temperature of the TMS to that of the oral cavity in this custom-made set-up. This fact could have had an impact on PVA rigidity, which is temperature sensitive.

In our early experiments, we identified that the results were strongly impacted by the amount and spreading pattern of the food deposited on the surface of the TMS. In this feasibility study, it was necessary to find robust and reproducible experimental conditions to be able to compare products and operating conditions. After a long series of preliminary tests, a protocol was developed and applied very strictly with regards to food deposition and spreading. In each experiment, 0.25 g of the model food was deposited in the form of droplets on the surface of the TMS (see Figure 5b.2b). The sample was then spread over the TMS using a spatula until an even visual aspect was obtained (see Figure 5b.2c). Once this step was completed, the aluminum plate was brought in contact with the TMS until the target value of the normal load was reached. As a consequence, similar initial conditions of spreading were attained for all the experiments, regardless of the rheological complexity of the food type (e.g., viscosity, yield stress).

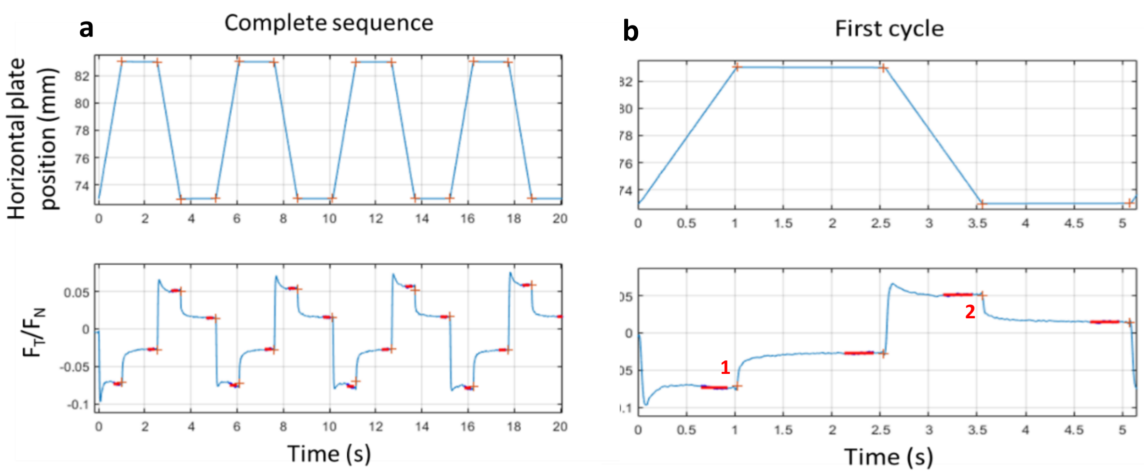
Although the set-up was designed for complex sequences of motion, in this study, we investigated a simple protocol based exclusively on the shearing motion of the horizontal stage (along the “x” axis) at constant velocity and a fixed position of the vertical stage (along the “z” axis) (Figure 5b.1). Once the target normal load had been achieved, the experimental sequence was launched. The experimental sequence consisted of four identical cycles of motion. Each cycle was composed of a 10 mm displacement of the horizontal plate in the “x” axis at a set velocity, a 1.5 s pause, a backward movement at the same velocity to return to the starting point, and another 1.5 s pause. It was necessary to incorporate a 1.5 s pause to separate the forward and backward movements but also to investigate the relaxation phenomena that may occur with these soft surfaces. At the end of the experimental sequence, the TMS was washed carefully with water to remove any residual food from the surface.

The experiments were performed with different applied normal loads (5, 10, 15, and 20 N with a shearing velocity of 10 mm/s) and different shearing velocities (5, 10, 20, and 30 mm/s at a normal load of 10 N). The normal load values were divided by the palate’s surface area in contact with the TMS to convert them into normal stress values (range of 4.5 kPa to 18 kPa). The choice of these normal stress values was made keeping in mind the maximum isometric tongue pressure (50 kPa on average) reported by Alsanei, Chen, & Ding (2015). However, as this study employed a semisolid food matrix, which does not require high levels of tongue contraction, the chosen values were deliberately kept low when compared to those reported in the above-mentioned article.

### 5b.2.4. Data processing and set-up validation

The raw force data (tangential and normal) obtained from the sensor were run through a low-pass filter (frequency threshold: 100-Hz) to improve the signal-to-noise ratio while respecting the sensor's response time. Then, the filtered force values and the horizontal plate displacement data for each experiment were fed into a custom-built MATLAB program (The MathWorks, Massachussetts, USA). For each experiment, the four cycles (forward and backward) of the movement sequence were studied. The program made it possible to visualize the variation in normal force and tangential force throughout the sequence of motion.

For each cycle, friction coefficient values were quantified by synchronizing the following data (represented in two plots; Figure 5b.3a): (i) the position of the horizontal plate (the one holding the TMS) over time and (ii) the ratio of tangential force ($F_T$) to normal force ($F_N$) over time. With the stage displacement data, time windows (0.3 s) before the movement stopped (marked as 1 and 2 for the first motion cycle, in Figure 5b.3b) were isolated. These time windows were chosen to be in the steady domain of the variation in friction coefficient values. Then, to obtain an overall friction coefficient value for one cycle, the values of the $F_T/F_N$ ratio (for time windows 1 and 2) were averaged over each cycle. The obtained friction coefficient discussed in this study thus does not take into consideration the potential differences between forward and backward phases. All the friction measurements were estimated using at least six replicates.




**<u>Figure 5b.3:</u> (a) Variation in horizontal plate displacement and the FT/FN ratio over time for an experimental sequence—the red highlights the time windows (0.3 s before the movement stops) used for calculating the friction coefficient at steady state and (b) variation in horizontal plate displacement and the FT/ FN ratio over time exclusively for the first cycle, where the windows marked 1 and 2 were used to calculate the friction coefficient for the cycle.**

Statistical tests were performed using XLSTAT (Addinsoft, Paris, France) to determine whether significant differences existed among treatments. T-tests were used when the measurements were normally distributed, and Kruskal-Wallis tests were used when they were not. Multiple comparison tests were performed to identify which groups differed significantly. The alpha level for the statistical tests was 0.05.

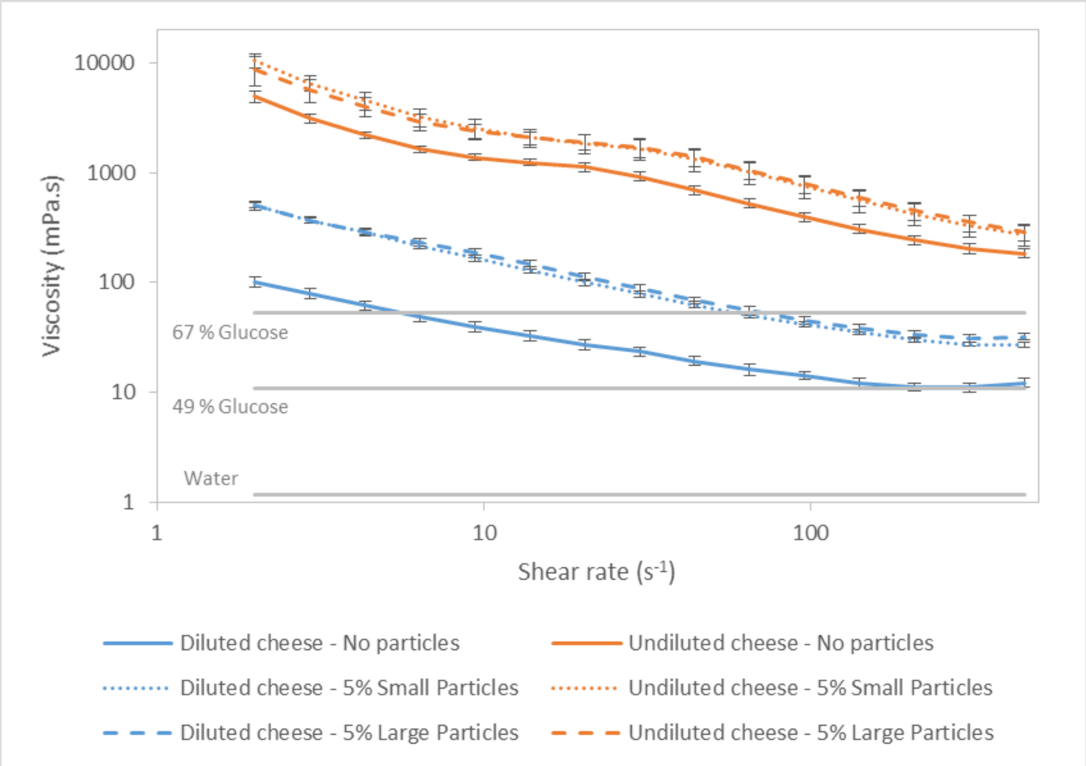
## 5b.3. Results and discussion

The feasibility, reliability and robustness of the set-up presented in this study were tested by measuring how the friction coefficient values were affected by changes in food properties (viscosity, particle presence), TMS properties (surface roughness, bulk rigidity) and operational parameters (normal stress, shearing velocity).

### 5b.3.1. Role of food matrix viscosity and heterogeneity

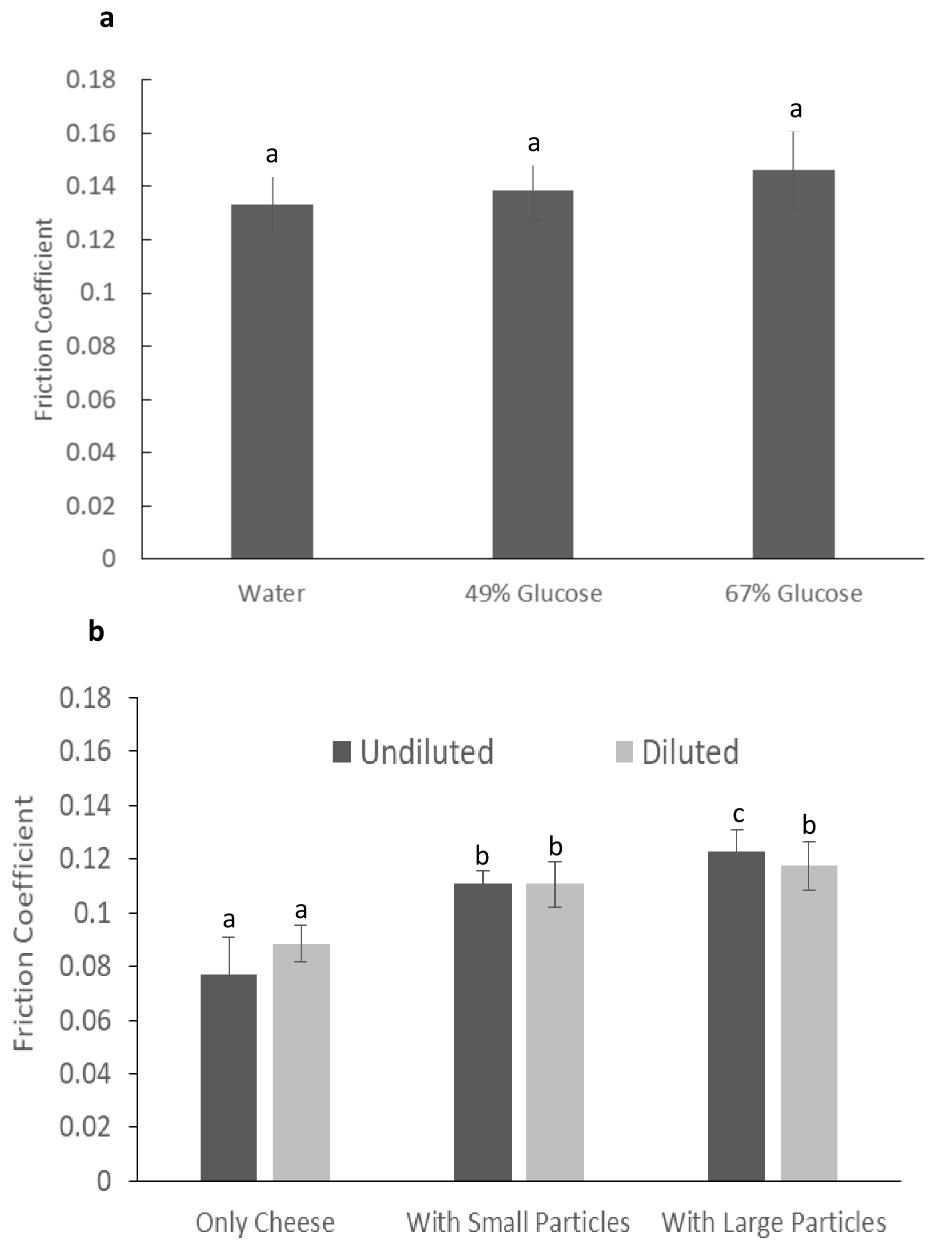
When surfaces are lubricated, as in the oral cavity, friction phenomena greatly depend on the properties of the lubricant (saliva or the food itself). The Stribeck curve shows that lubricant viscosity is one of the important factors governing friction (Chen & Stokes, 2012). However, in the case of a food system, the matrix is more complex because it contains heterogeneities that, in turn, impact the overall friction mechanism. Therefore with this developed set-up with parallel and planar surfaces, it was of utmost importance to investigate how friction forces at the interface were affected by the synergistic role of food viscosity and heterogeneity.

First, viscosity flow curves were studied for the different cottage cheese types (diluted or undiluted; with or without particles; Figure 5b.4). As expected, all the cottage cheeses showed shear thinning responses with increases in shear rate, confirming their non-Newtonian behavior. Also, the undiluted cottage cheese displayed much higher viscosity than did the diluted cottage cheese throughout the experiments. The presence of particles in both the diluted and undiluted cottage cheeses led to an increase in viscosity. However, particle size did not seem to have any impact (given the overlap in standard deviation). Moreover, diluting the cottage cheese had a much more pronounced effect on viscosity than did incorporating suspended particles of any size. It should also be noted that, even when shearing took place, the mean viscosity of the undiluted cottage cheese (both with/without particles) remained far above the mean viscosity of the glucose solutions (plotted as a reference gray line). However, the diluted cottage cheese (without particles) displayed a drop in viscosity at lower shear rate ($< 10\ s^{-1}$), matching the mean viscosity of the 67% glucose solution; it further declined at high shear rate, reaching a mean viscosity comparable to that of the 49% glucose solution. A similar trend was seen for the diluted cottage cheese with particles, although its mean viscosity remained far above that of the 49% glucose solution.




**<u>Figure 5b.4:</u> Flow curves for the different types of cottage cheese, with the mean viscosity values of the glucose solutions plotted as a reference.**

Given the confirmation of the distinct rheological behaviors of the cottage cheese types, tongue-palate shearing tests were performed on rough and soft TMSs to investigate the friction behavior of the tribological system. For the friction measurements, normal stress and shearing velocity were fixed at 9 kPa and 10 mm/s, respectively. Mean friction coefficient values were obtained for the glucose solutions (Newtonian fluids; Figure 5b.5a) and the cottage cheese types (non-Newtonian fluids; Figure 5b.5b). The friction coefficient values of the three glucose solutions were not significantly different; the solutions did, however, differ in viscosity. The cottage cheese types also displayed significant differences in viscosity. In contrast, there was no significant difference in the friction coefficient values of the diluted versus undiluted cottage cheese. However, relating friction coefficient and viscosity values is difficult in the case of non-Newtonian fluids, as the gap between the tribo-pair in the set-up changes over the course of shearing, making it impossible to accurately estimate the corresponding value of the shear rate.




**<u>Figure 5b.5:</u> (a) Friction coefficient values for the glucose solutions (0%, 49%, and 67%) and (b) friction coefficient values for the different types of cottage cheese (undiluted/diluted, with/without particles — small and large). Normal stress and shearing velocity were 9 kPa and 10 mm/s, respectively. The error bars represent the standard deviations across six replicates. The differences in letters indicate significant differences between groups.**

Interestingly, the presence of particles in the cottage cheese clearly had a significant impact on the friction coefficient values and proved to be a key governing factor (independent of the impact of particle presence on cottage cheese viscosity). Water absorption by the particles (and the resulting changes in the free water content of the cottage cheese) could hypothetically have explained the differences in lubricity among cottage cheese models. However, our results provide little support for this hypothesis (Figure 5b.5b). In the cottage cheese without particles, dilution barely affected the lubrication properties even with the drastic drop in viscosity (similar friction coefficient values). According to the particle supplier, the maximum water absorption capacity of the microcrystalline cellulose particles used in this study is less than 2.7 g/100 g. Moreover, their concentration in the cottage cheese types was only 5% (w/w). As a consequence, the ability of these particles to absorb water from the cottage cheese likely had negligible effects in comparison to the dilution treatment.

The above results thus demonstrate that the set-up described here can measure steady and repeatable friction forces for model foods that vary in rheological behavior. The friction coefficient values for the undiluted cottage cheese without particles fell within the range of values reported in Laguna et al. (2017), which measured the friction coefficient values of low-fat cream cheese and yogurts that were undiluted or diluted with artificial saliva (1:1 ratio) using a ball-on-disk traction tribometer (PDMS tribo-pair).

### 5b.3.2. Role of surface roughness

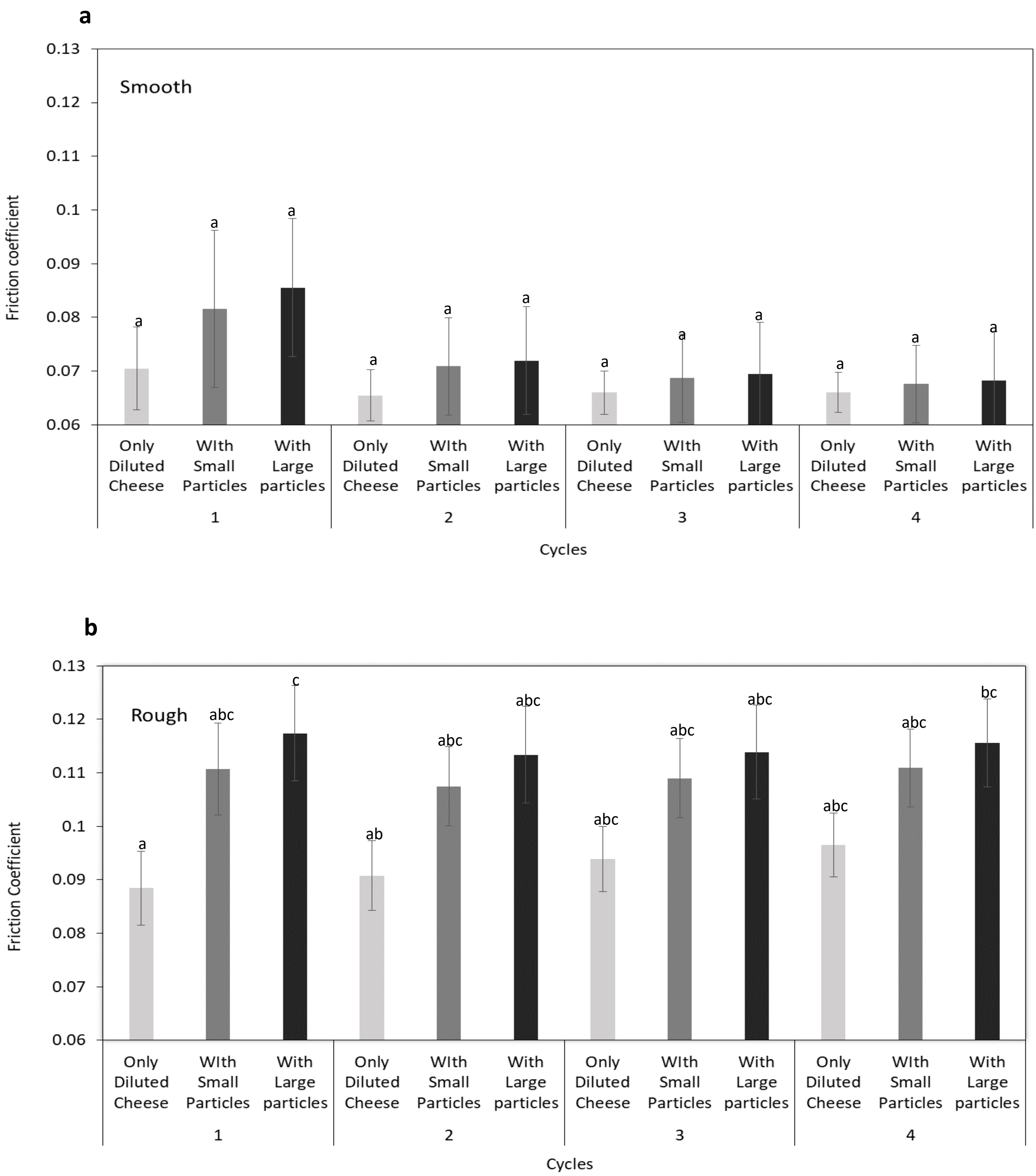
Surface roughness is also known to greatly impact friction mechanisms. Tallian (1967) introduced the parameter $\Lambda$, which is defined as the ratio between the thickness of the lubricant film and the roughness of the moving surfaces. This parameter expresses the ability of the lubricant film to prevent direct contact between two surfaces and is directly related to the lubrication regime ($\Lambda > 3$ for elastohydrodynamic and hydrodynamic regimes, $1 < \Lambda < 3$ for mixed lubrication, and $\Lambda < 1$ for boundary lubrication). Nonetheless, the roughness of the tongue remains sparsely accounted for in oral tribological studies. Bongaerts, Fourtouni, & Stokes (2007) rightly pointed out that, in the case of soft surfaces, surface asperities may become deformed and result in a complex elastohydrodynamic regime of lubrication. Therefore, taking into consideration TMS roughness was one of the key requirements for making the set-up as similar as possible to a real oral surface.

In this study, we compared two surface profiles, rough and smooth, which were created by molding TMSs with and without sandpaper, respectively. The rigidity of both model tongues was similar (soft: 50.17 ± 1.46 kPa). Three types of cottage cheese were used to examine the effects of surface roughness: diluted cottage cheese without particles, with small particles, and with large particles. Normal stress and shearing velocity were kept constant at values similar to those in the experiments described in section 3.1 (9 kPa and 10 mm/s, respectively).

The means and standard deviations of the friction coefficient were measured for the three cottage cheese types on smooth and rough TMSs (Figures 5b.6a and 6b). There was an evident impact of surface roughness on friction coefficient values (p-values < 0.0001 for the comparisons of each cottage cheese type on rough vs. soft TMSs). For all three cottage cheese types, higher friction coefficients were obtained with the rough versus smooth TMSs. The results thus suggest that the food is less able to prevent contact between the TMS and the palate in the case of the rough TMSs than in the case of the smooth TMSs. One possible explanation for this observation is that the introduction of asperities to the rough TMSs may have led to the more efficient spreading of lubricant, resulting in a thinner gap between the tribo-pair.

Additionally, although the trend was not statistically significant, the value of the friction coefficient seemed to be constant with respect to the number of shearing cycles for the rough TMSs but showed a decrease from the first to second cycle for the smooth TMSs. A more effective spreading of the lubricant film on the rough surface could once again explain this pattern. The initial application of normal stress could have maximized the spreading of the lubricant on the rough surface, and, consequently, the shearing cycles that followed would have had no further impact on film thickness, resulting in a constant friction coefficient value. On the other hand, on the smooth surface, the application of the same level of normal stress might not have led to maximal spreading, which was later achieved by the first shearing cycle.

It is also worth noticing that, compared to the smooth surface, the rough surface made it possible to slightly better segregate the cottage cheese types with or without particles. Also, there was a trend for friction coefficient values to be slightly higher for cottage cheese with large particles than for cottage cheese with small particles, even if this difference was not statistically significant at a given particle concentration, normal load, and shearing velocity. However, it could be that there are certain combinations of operational parameters (normal stress and shearing velocity) and TMS properties (roughness and rigidity) that could better segregate the results in relation to particle size.



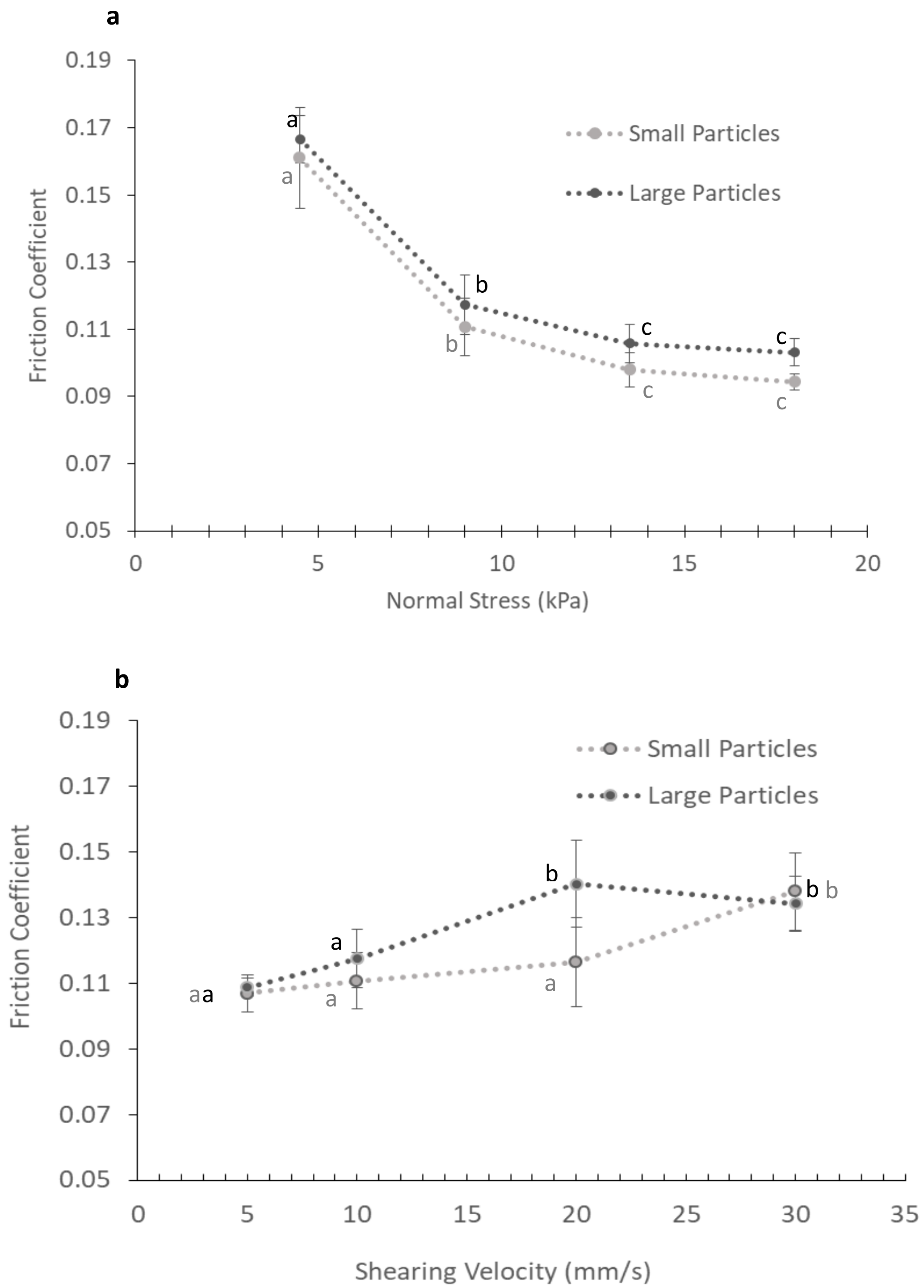

**<u>Figure 5b.6:</u> Friction coefficient values for the diluted cottage cheese with or without particles (small or large) on soft TMSs with (a) a smooth surface profile and (b) a rough surface profile. Normal stress and shearing velocity were 9 kPa and 10 mm/s, respectively. The error bars represent the standard deviations across six replicates. The differences in letters indicate significant differences between groups. The p-values for the comparisons of the results for the two cottage cheese types on the soft and rough profiles were all < 0. 0001.**

### 5b.3.3. Role of operational parameters: normal stress and shearing velocity

When studying the feasibility of this tribological set-up, it was imperative to investigate the evolution of friction forces in response to variation in normal stress and shearing velocity. The Stribeck curve can clarify how these parameters (in tandem with lubricant viscosity) can govern the lubrication regime of the system by impacting lubricant film thickness.

When studying the impact of normal stress, the experiments were performed with a rough artificial tongue with a rigidity of 50.17 ± 1.46 kPa (soft) and diluted cottage cheese containing small versus large particles. The means and standard deviations of the friction coefficient were measured for six replicates of both cottage cheese types across a normal stress range of 4.5–18 kPa and at a constant shearing velocity of 10 mm/s (Figure 5b.7a). It is evident from these results that increases in normal stress (from 4.5 kPa to 13.5 kPa) led to a significant decline in friction coefficient values. However, for the highest normal stress value, the decrease in the friction coefficient was not significant, suggesting a nonlinear response. This trend was observed for both cottage cheese types. A similar dependency of the friction coefficient on the normal load was also reported by Chojnicka, De Jong, De Kruif, & Visschers (2008) for two different protein dispersions (ovalbumin and whey protein isolates). These trends reflect that the system can occur somewhere between the hydrodynamic and the mixed lubrication regimes as contact between the palate and the TMS becomes more pronounced with increased normal stress. However, to identify the exact lubrication regime, the complete Stribeck curve needs to be built. It is also worth noting that, with the increase in normal load, the reproducibility of the results improved considerably. An explanation is that, when the normal load is increased, there is an improvement in the signal-to-noise ratio of the measured forces.

Additionally, the effect of variation in shearing velocity at a given normal stress was also studied. Results were obtained on the rough surface for diluted cottage cheese containing small and large particles at a normal stress of 9 kPa and shearing velocities of 5, 10, 20, and 30 mm/s (Figure 5b.7b). Here, the difference in the friction coefficient values at the maximum and minimum velocities was significant: values were higher at 30 mm/s than at 5 mm/s. However, increases within the intermediate velocities did not have a statistically significant effect. It may be that, at the given normal stress, higher velocities need to be tested.




**<u>Figure 5b.7:</u> Friction coefficient values for the diluted cottage cheese with small or large particles on the soft or rough TMSs under the following conditions: (a) variable normal stress and a fixed shearing velocity of 10 mm/s and (b) variable shearing velocity and a fixed normal stress of 9 kPa. The error bars represent the standard deviations across six replicates. The differences in letters indicate significant differences between groups.**

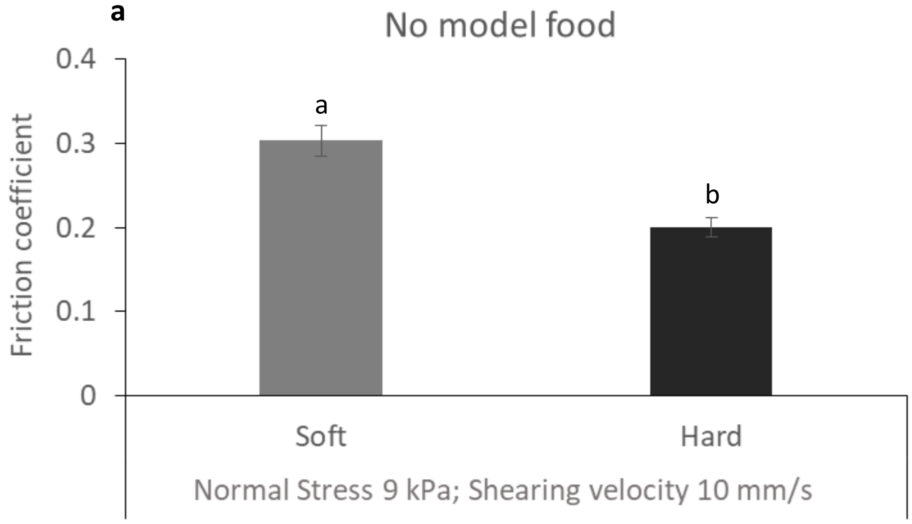
### 5b.3.4. Role of TMS rigidity

During the complex in vivo scenario of food oral processing, tongue rigidity and shearing velocity shift in order to change friction forces and thus better perceive certain textures. The PVA-based TMSs in this study provided an opportunity to compare friction measurements for two different rigidity values. A hard TMS (100.78 ± 2.12 kPa) was therefore introduced into the experimental design, and its results were compared to those for the soft TMS (50.17 ± 1.46 kPa). The bulk rigidity values of both the soft and hard TMSs were similar to those reported by Ishihara et al. (2013) for relaxed and contracted human tongues. Both TMSs had rough surface profiles.

To begin with, experiments were performed on both the TMSs without any food. Normal stress and shearing velocity were 9 kPa and 10 mm/s, respectively. It was found that having lower rigidity imparted higher friction force (Figure 5b.8a). In the case of the non-lubricated soft surface, the adhesion of the hydrogel to the aluminum probe might have created a backward force, resulting in a higher friction force. For the lower rigidity surface, the deformation of the asperities was higher under compression, which, in turn, resulted in a larger contact area and thus higher adhesion between the TMS and the palate.

Then, experiments were performed using diluted cottage cheese containing small and large particles. Two different values of normal stress (9 kPa and 18 kPa) and shearing velocity (10 mm/s and 20 mm/s) were tested. For the sake of simplicity, only the results pertaining to the cottage cheese with small particles are presented because the cottage cheese with large particles showed similar trends (Figures 5b.8b and 8c). The friction behavior of the two TMSs under lubricated conditions was opposite to that observed under non-lubricated conditions (Figure 5b.8a). The hard TMS led to higher friction coefficient values than did the soft TMS, regardless of the level of normal stress or shearing velocity. This reversal in friction behavior between situations with and without food is clearly due to the lubrication properties of the diluted cottage cheese. In the absence of the food (i.e., dry contact), friction was driven by adhesion, which was greater for soft TMSs, as explained above. In contrast, during lubricated contact, the cottage cheese restricted absolute contact between the aluminum palate and TMSs and also altered the adhesion properties of the hydrophilic PVA surface. Higher deformation of the soft TMSs (compared to the hard TMSs) also made it easier to evenly spread the lubricating film, preventing any dry adhesive contact between the TMS and the palate.

a

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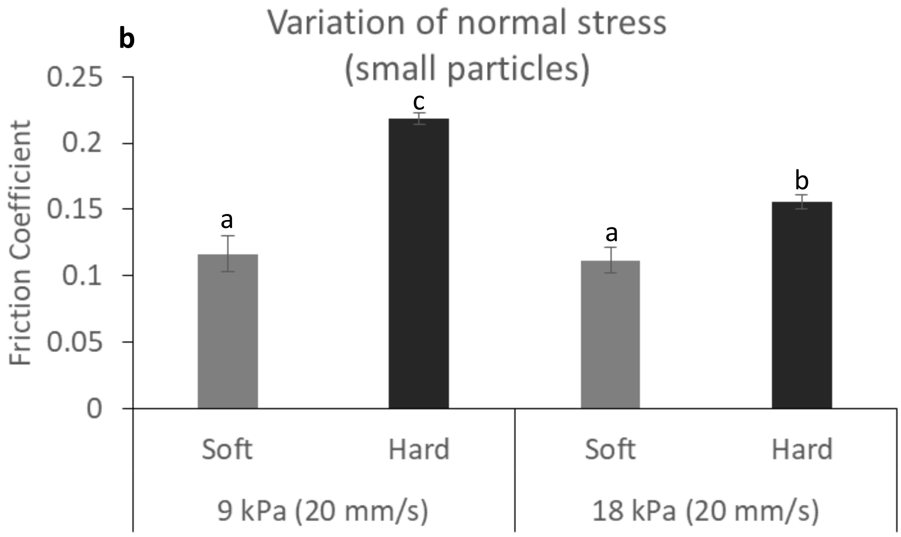


**<u>Figure 5b.8:</u> (a) Friction coefficient values on hard and soft TMSs with rough surface profiles when no food was present; (b) friction coefficient values on hard and soft TMSs with rough surface profiles when using diluted cottage cheese with small or large particles under conditions of variable normal stress; and (c) friction coefficient values on hard and soft TMSs with rough surface profiles when using diluted cottage cheese with small or large particles under conditions of variable shearing velocity. The error bars represent the standard deviations across six replicates. The differences in letters indicate significant differences between groups.**

Furthermore, the responses to changes in normal stress or shearing velocity were similar to those observed in the previous sections. Increases in the normal load and the shearing velocity led to decreases and increases, respectively, in the friction coefficient values. These results again confirm that variation in such operational parameters could govern the lubrication regime and may sometimes enable the transition from one regime to another. Furthermore, it was also evident that changes in the friction coefficient values were more pronounced in the case of the

TMS with higher rigidity. Apart from this higher rigidity, the freezing and thawing cycles of the PVA might also have influenced the surface asperities (especially in terms of asperity height), which could be one of the possible explanations for the higher friction coefficient values.

Finally, with the exception of surface roughness, all the physiological properties mimicked here (rigidity, normal load, shearing velocity) are highly likely to change based on how the consumer adapts his/her oral strategy to a food's properties and how he/she manipulates food in the mouth. The results of this study show that varying these parameters greatly affected the friction coefficient values. Consequently, this set-up opens the door to exploring the relationships between oral strategies and their consequences for friction behaviors. Finally, this set-up with deformable PVA could be useful for any in vitro research examining the dynamics of texture perception.

## 5b.4. Conclusions

In this study, a tribological set-up was successfully developed to achieve more realistic oral conditions for investigating tribological mechanisms during food oral processing. The set-up made it possible to use deformable tongue-mimicking samples (TMSs) made with polyvinyl alcohol (PVA), which facilitated the imprinting of a particular level of surface roughness and the management of bulk rigidity. Moreover, like with any other conventional tribological equipment, it was possible to apply different ranges of normal stress, shearing velocity, and displacement sequences.

The feasibility and reliability of the set-up was investigated by examining the dynamics of different food types and TMSs in response to variation in different operational parameters such as normal stress and shearing velocity. For each set of test conditions, friction coefficient values were reproducible. The results obtained for the cottage cheese containing suspended cellulose microcrystals highlighted the importance of accounting for the complexity of tongue rigidity and roughness to better comprehend the frictional phenomena involved. Parameters such as the normal stress or the shearing velocity between the tongue and the palate were shown to be important drivers of variation in the friction coefficient at the tongue-food interface. However, these parameters were also found to be tightly linked to TMS properties like roughness and rigidity. The addition of microscale heterogeneities to the cottage cheese did result in an increase in friction coefficient values, but it was also interesting to note that the results did not segregate based on particle size for a given operational parameter. This information can further be used to hypothesize that there could be unique combinations of normal stress and shearing velocity for a given TMS that might result in the segregation of results based on different types of heterogeneities (size or shape). However, such work would require a more detailed experimental design, which is a goal for future research.

The friction coefficient was the main physical property analyzed in this study. However, the force sensor used in the bio-mimicking set-up offers many other opportunities for understanding the physical phenomena occurring between the tongue and the palate during shearing motion. Examining the relaxation phenomena that take place right after the displacement steps could reveal how the asperities of the tongue surface behave and recover their initial shape after having been subjected to shear stresses, with respect to the presence of a lubricating film or food bolus. Another objective for future work will be to quantify and to investigate the origin of the possible differences in friction coefficient between forward and backward motions (potentially related to the level of residual tangential stress at the end of relaxation steps). Our study represents the first instance of PVA being used as a model tongue; this approach shows promise but must be improved over the course of future studies. However, to better mimic the topology of the human tongue, it will be necessary to focus on new techniques for molding surfaces and analyzing surface profiles. Real or artificial saliva could also be used to mimic lubrication and friction mitigation in the mouth. Also, in future studies, a larger degree of variation in normal stress and shearing velocity should be explored to better establish the relationship between the friction coefficient and the Stribeck curve. Finally, the set-up itself can be modified to perform more realistic motions (e.g., oscillatory, translational) and to allow more robust control and automation.

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# VI
# General Discussion

The results obtained during the course of this PhD project have allowed us to further advance towards better understanding the mechanical phenomena that occur at the tongue-food interface during the oral processing, by not only better adapting mechanical characterization methods (uniaxial compressions with texture analyzer) with respect to oral conditions, but also by developing a completely new biomimetic setup. The objective of this general discussion chapter is to discuss how the different objectives of this thesis contribute to the field of food oral processing in a holistic way.

## 6.1. Towards more realistic in vitro setups

**Tongue deformability and surface roughness: key parameters for understanding food compression during food oral processing**

As previously underlined in the literature review, the need to perform the mechanical characterization of food in physiologically relevant systems is paramount for understanding resultant texture perception. The two biomimetic setups used in this study were aimed towards this goal. To begin with, the setup developed by Mantelet (2020a and 2020b) was modified, by introducing artificial tongues made of polyvinyl alcohol which helped us to modulate the rigidity of the tongue model in addition to the surface roughness (Chapter 3). The use of PVA based tongue mimicking surface was also extended to the newly developed oral tribology setup (Chapter 5a and 5b). The use of deformable tongue surfaces is growing in literature, and researchers have used different polymer based tongue models for understanding the food breakdown by uniaxial compression. However, the role of surface roughness is often ignored. In this thesis (Chapter 3), it was observed that the deformation of the PVA based artificial tongue model (or the exerted force) during the compression of gels were not identical for two different surface roughness profiles despite having same rigidities (Figure 3.6). Similarly in tribological studies, the use of oral mimicking surfaces has gained attention and is discussed in the literature review. However, here the focus of the scientific community is mostly on mimicking the surface roughness of the tongue. We are well aware of the fact that the tongue's rigidity is modulated continuously for better manipulation of the food, and in our results from chapter 5 (Figure 5b.8) we notice that bulk rigidity of the tongue can have significant impact on friction forces. Hence both of these examples underscore the need to understand the synergistic role of tongue's rigidity and roughness in food manipulation and the resultant perceptions of texture.

**Ultrasound time of flight: a relevant parameter to assess the tongue and food deformability**

The introduction of ultrasound based monitoring of the tongue-food system and its interface also brings an added value to the thesis project. This novel method of characterization performed in real time, allowed to define key ultrasound parameters for analyzing the tongue-food system. Building upon the method by Mantelet (2018), and taking into account the deformability of the tongue model, the time of flight of echoes reflecting from tongue-food interface (ToF) was identified in addition to the apparent reflection coefficient at the interface. In chapter 3, with the help of ToF values, the deformation of the model tongue was estimated in real time during the compression of food, which is very original. The idea of measuring the tongue deformation is very important in context of texture perception, as it can help to imagine indirectly the transfer of stress to the mechanoreceptors (in real life scenarios).

Moving a step forward, in chapter 4, the measurement of the tongue deformation by the ToF was extended to food gels as well. In this part, more complex bilayer gel models were used to push the scope of using ToF method in understanding the response of different mechanical entities (tongue and bilayer model gels) under a uniaxial compression. The vision behind this approach was to comprehend the mechanical phenomenon behind the perception of heterogeneity. The traditional texture characterizing instrument can only provide an overall physical measurement of such composite systems, and fail to give information about the mechanical response of each structural entity. However, the developed ultrasound method answers to this problem. By monitoring the ToF from different interfaces the deformation of each layer can be easily estimated. Although, some adaptation in ultrasound transducer frequency and signal processing was done to fine tune our measurement for the complexity of bilayer gels. In chapter 4, we observed (Figure 4.5 and 4.6) how the mechanical properties (Young's modulus) of each layer, the polymer type (elastic gelatin or brittle agar) and position (top or bottom) had a considerable impact on the overall deformation of the tongue food system.

**Assessment of interface and contact mechanism between food and tongue with apparent reflection coefficient**

The apparent reflection coefficient (R*), as already highlighted by Mantelet et al. (2020a and 2020b) to describe the interactions between the tongue and the food, was found very useful. In the case of an ideal coupling (when there is no air trapped at the interface between the food and the artificial tongue), the reflection of the ultrasound waves at the interface is governed by the difference in acoustic impedance between the two media. In the case of the feasibility studies carried out in the framework of Mathieu Mantelet's thesis, the artificial (non-deformable) tongue was made of PVC. There was then a difference in acoustic impedance between the tongue and the gels, with reflection coefficient values in the case of perfect coupling reaching 30-35% (Mantelet et al., 2020a). This means that even when the coupling is ideal, a significant part of the ultrasound beam energy is reflected by the interface. By choosing to change the material to take into account the deformability of the tongue and thus get closer to the mechanical and acoustic properties of a real tongue, we knew that this acoustic impedance gap would decrease drastically and there was thus a significant amount of uncertainty as to the ability of the method to keep its potential. Indeed, we did observe, in the case of the coupling between a PVA tongue and $Ag_{0.3}$ gel which is both the softest and the most prone to release water (and thus the one that leads to the most ideal coupling, without the presence of air at the interface), that the reflection coefficient was close to 0%. This indicates that in the case of an ideal coupling, the amplitude level of the tongue-food interface echo is very low, or even null. In spite of these constraints, we had to make this improvement as it is an essential condition for the success of the method and to continue the quest for its deployment in *in vivo* conditions. However, we knew that we were not going to hit a dead end thanks to preliminary tests that had been carried out in the framework of Mathieu Mantelet's thesis. The use of an ultrasound imaging probe had then allowed us to visualize the tongue interface during the oral processing when the agar and gelatin gels were consumed. These tests suggested that in "real life" conditions, the coupling is not ideal and that the combined properties of the gels and the tongue lead to air entrapment at the interface. The work carried out during the thesis confirmed these hypotheses. It led us to evolve the signal processing methods in order to have robust programs for echo detection, despite an unfavorable signal-to-noise ratio compared to the feasibility studies carried out with artificial PVC tongues. These points were all the more important as they conditioned the accuracy of the quantification of the two ultrasound parameters studied during the thesis. This was also very encouraging as it opens the possibility of employing ultrasound in vivo where the body tissue has almost similar acoustical impedance with that of most foods and bolus. As discussed by Mantelet et al. (2020a and b), the variation of R* indicates the acoustical coupling of the tongue and the food during compression, and which has shown to be dependent on the water released into the interface or the molding of the tongue asperities by the gel itself. The similar observations were recorded with PVA based model tongues. Moreover, the role of rigidity was also significantly noticed. As we know, the rigidity of the tongue affects the deformation of food and its own surface asperities resulting in variation of coupling phenomenon at the interface. Hence this real-time, non-invasive ultrasound based monitoring of the tongue-food interface proved to be very effective in bringing new insights about the contact mechanism and its progression during the compression. It has enabled us to highlight the importance of various parameters that can influence physical phenomena, such as coupling at the interface or the deformation of the tongue during simple uniaxial compressions.

However, it is obvious that the oral manipulation of food by the tongue is not limited to simple compression movements. On the contrary, shearing movements are very important for the stimulation of the mechanoreceptors, especially when rubbing against the palate. The development of a new biomimetic system was therefore necessary to incorporate these complex shearing motions.

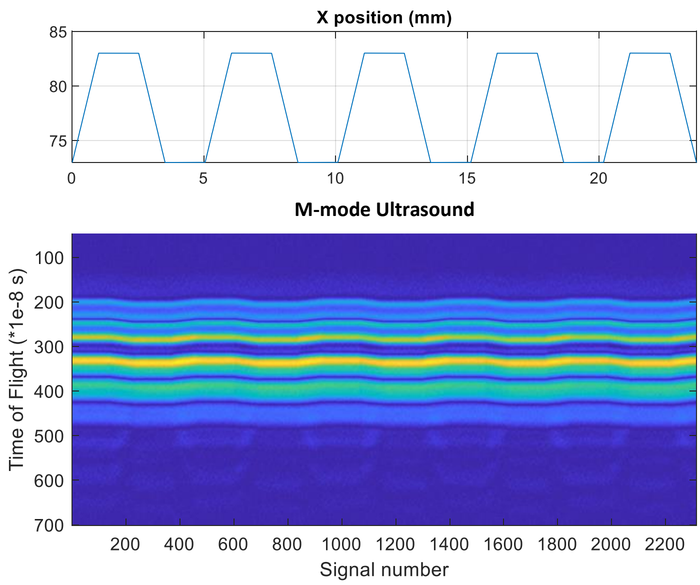
**The new setup with shearing motions**

As described in Chapter 5a, the prototype was built by assembling commercially available structural parts but also fabricating custom made. PVA based artificial tongue models were employed here as well. The setup was capable of applying desired normal load, and perform shearing with desired velocities with different motion sequences. The dimensioning of the sensors and the various mechanical elements of the system was thus carried out by taking into account the oral characteristics (speed of displacement of the tongue, stress exerted during oral processing…). A human-machine interface was also entirely designed in order to control the system and to acquire the data from the different implemented measurement channels. The developed setup offers a wide range of possibilities because of the sequences of movements it is able to perform (while varying the properties of the artificial tongues), but also because of the different measurements it allows (three-axis force sensor, accelerometers, ultrasonic sensors) and that can also be further extended thanks to its adaptability.

Given the novelty of the system, it was necessary to continue our logic of gradual progression, by putting ourselves in an experimental framework that would allow certain comparisons with literature. The main novelty of the system being to be able to work in shear, the choice was thus made to consider protocols of movements close to the sequences which can be carried out in tribology. This is how we arrived at the protocols of back and forth movements in shear, imposed under certain fixed conditions of normal loading and shear rate. We performed the first proof of concept study (see chapter 5b) focusing on the friction forces associated with lubrication induced by the presence of liquid food.

For this first proof of concept study, water, glucose solutions and cottage cheese (diluted/undiluted and with/without cellulose particles) were used. The setup as aspired, helped to study the effect oral complexities (surface roughness and rigidity of the tongue) and operational parameters (normal stress and shearing velocity) on evolution of friction force (discussed in details in chapter 5b). The analysis of the frictional forces has thus allowed a first validation of the interest and potential of the new biomimetic system to study issues related to the oral processing.

Moreover, as we have already said, the new system also embeds ultrasound sensors, as used in the first part of the thesis. However, the application of ultrasonic methods to the experimental conditions considered in the feasibility study described in chapter 5b could not be done in a simple and straightforward manner. The first reason being that when working with liquid foods, the mechanical coupling at the tongue almost always proves to be ideal and the tongue-food interface does not offer as energetic acoustic response as has been quantified so far. The second reason is that when working with a food that forms a very thin lubricating film (around 100µm), the sensor frequencies that can be used do not allow to distinguish the tongue-food interface from the food-palate interface. Finally, the third reason is that by considering only shear movements, the amplitude level of the time-of-flight variations is very low. To illustrate, figure 6.1 shows the ultrasonic signals acquired during one of the test sequences associated with the work performed in chapter 5b. Similar to the uniaxial compression setup, an ultrasound sensor was placed underneath the tongue. The ultrasonic signals are shown in M mode (Figure 6.1).




**<u>Figure 6.1:</u> Variation of time of flight during the tongue-palate shearing experiment (in the absence of food) w.r.t. to movement of horizontal stage (one which holds the tongue).**

This is a visual representation of the succession of all the ultrasonic signals acquired over time during the test sequence. The abscissa axis corresponds to the time elapsed during the test. The ordinate axis corresponds to the propagation time of the ultrasonic waves. The image represents the amplitude of the signals, coded by a color scale from blue to yellow. The upper part of the image (in blue) corresponds to the time elapsed before the ultrasound wave reflects at the interface with the upper surface of the tongue (which may be in contact with either the food or the palate, the two being indistinguishable at this frequency). As soon as the wave is reflected, echoes begin to be perceived, the shape and duration of which are complex, but the first peak of which provides an indication of the level of deformation of the tongue throughout the test. We notice that when the plate is stationary, the ToF of echoes remains constant. On the contrary, when the plate is in motion the ToF evolve as well.

The ToF variations we observe here are primarily associated with errors in parallelism between the palate surface and the axis of movement of the shear translation stage. While it is interesting to quantify this error (which in this case represents a deviation of about 100 µm in the vertical direction over an amplitude of 10 mm of horizontal displacement), we believe that ultrasonic methods still have a very interesting potential on this device. First, it will still be possible to work with semi-solid foods (which will cause coupling issues and which will keep a sufficiently important thickness to distinguish echoes). Then, it will be possible in the short or medium term to consider sequences of movements combining compression and shear (during which it will be possible to distinguish the interfaces, at least at certain moments of the test sequences). Moreover, by increasing the pulse recurrence frequency, it could also be possible to detect fluctuations in the time of flight at very short times, which could reflect vibratory events occurring when rubbing the tongue against the palate. This is a work that will have to be further developed after this thesis project.

## 6.2. Finding the sensory links

As argued in the context of this project, the efforts done in characterizing the food properties for understanding texture perception should be done in physiologically relevant systems, for better estimation of the sensorial outcomes. In this PhD project we have tried to develop setups which can perform physical measurements in conditions which are closer to oral conditions, but the question is whether these parameters can be related to sensory perceptions.

**Sensory perceptions of model gels v/s ultrasound parameters**

To begin with uniaxial compression of gels, this work that we started, was based on a sensory analysis study carried out during Mathieu Mantelet's thesis. The eight model food gels which were used in chapter 3 of this thesis were also used for sensory analysis study by Mantelet (2018).

Texture profile method was used for these gels and the protocol was designed to match the conditions under which ultrasound measurements have been performed. It thus allowed to evaluate the perceptions at two times during consumption, first when the food was deposited on the tongue and second after having performed a tongue-palate compression. The descriptors "Moist", "Slippery" and "Strength" (defined with the panelists as the ability to hold its shape without fracturing or collapsing during the deposit step) were evaluated when the gels were deposited on the tongue, followed by additional evaluation of descriptors like "Firm", "Elastic" and "Pasty" after tongue palate compression step. In Fig 6.2, we can see the ranking of the three attributes rated during the deposition of food on the tongue surface.

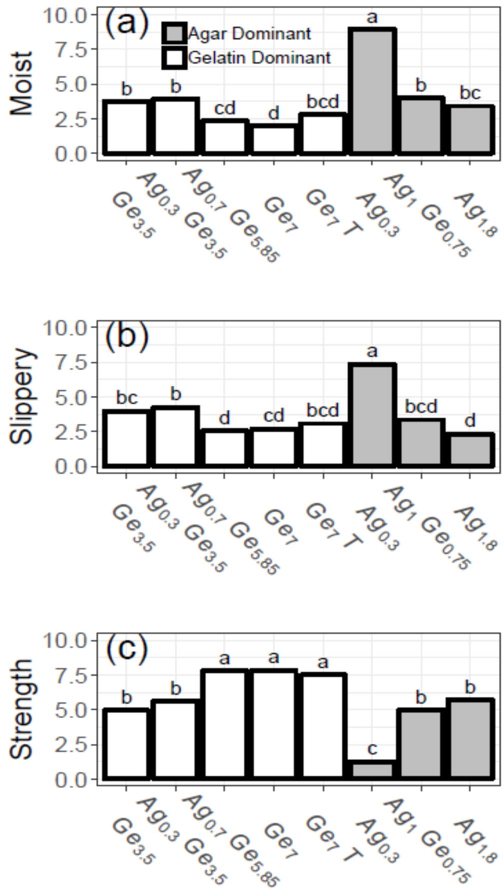


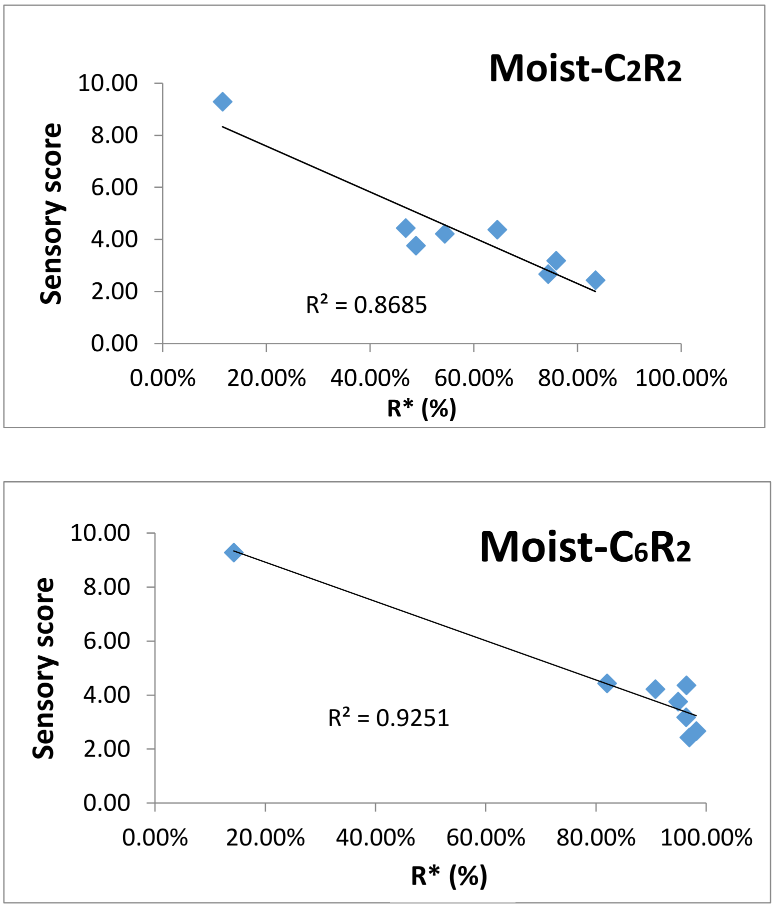

**Figure 6.2: Average sensory scores for the "Moist" attribute at the time of deposition on tongue. The letters represent the statistical groups to which the averages belong statistical groups to which the means belong.**

**Newman Keuls multiple comparison test, p<0.05%). (Mantelet, 2018).**

A first observation that can be made from these graphs is that from a sensory point of view, they confirm the singular character that we were able to identify from an instrumental point of view for $Ag_{0.3}$. The latter is perceived as being significantly more "moist" and "slippery", but with the lowest intensity for the "strength" attribute. These results are quite consistent with the results obtained using instrumental methods. It is known that $Ag_{0.3}$ is the sample that releases the most water by syneresis (resulting in low US reflection coefficient values), even without applying any mechanical stress other than its own weight. Therefore, the fact that it is perceived to be wetter is quite expected.

A second important observation that can be made when looking at these graphs is that although differences appear to be significant between some of the other gels considered in the study, they remain relatively small. Thus, even though we considered a total of eight gel references, the results of the sensory analysis show a 'product space' essentially composed of two groups ($Ag_{0.3}$ and the others), with little intermediate behavior in terms of perceived intensities. This is noteworthy because from an instrumental point of view (whether with classical characterization methods or with the ultrasound methods developed during the thesis), it was possible to describe a significant diversity of mechanical behaviors between all gels other than $Ag_{0.3}$. Although this needs to be confirmed, the results show that the methods we have developed provide a contrast between the mechanical behaviors of the gels that seems more important, when compared to the judges' ability to differentiate the gels.

Nevertheless, we tried to go a little further in linking the sensory and ultrasonic data. As discussed previously, the reflection coefficient provides insights about the coupling between the tongue-food. We plotted in figure (6.3) the reflection coefficient values (obtained before the commencement of the compression) and the ranking of 'Moist' attribute (ranked when the product was placed over the tongue). Similar correlation trends were also obtained for slippery and strength attributes. Interestingly, it was also observed that the points on the soft ATM ($C_2R_2$) were more spread than that of hard ATM ($C_6R_2$). There is no




**<u>Figure 6.3:</u> Representation of the trend observed between the sensory data (for the moist attribute, at the time of deposition on the tongue) and the ultrasound reflectivity (before commencement of compression) on two different ATMs.**

point here to spend much time discussing the strength of the correlation ratio here, as it is clearly dictated by the singular behavior of $Ag_{0.3}$. However, very interestingly, a greater dispersion of reflection coefficient values can be noted when the tests were performed on the $C_2R_2$ tongue (corresponding to a tongue in a relaxed state) than when they were performed on the $C_6R_2$ tongue (of similar roughness, but harder and thus corresponding to the tongue in a contracted state). This shows that the correlation relationships make more sense in the first case, which could shed new light. The more deformable the tongue, the more the mechanical behavior of the gels at the interface seems to contrast. This suggests that the relaxed state of the tongue may be more favorable for detecting contrasts in sensory perception between the gels.

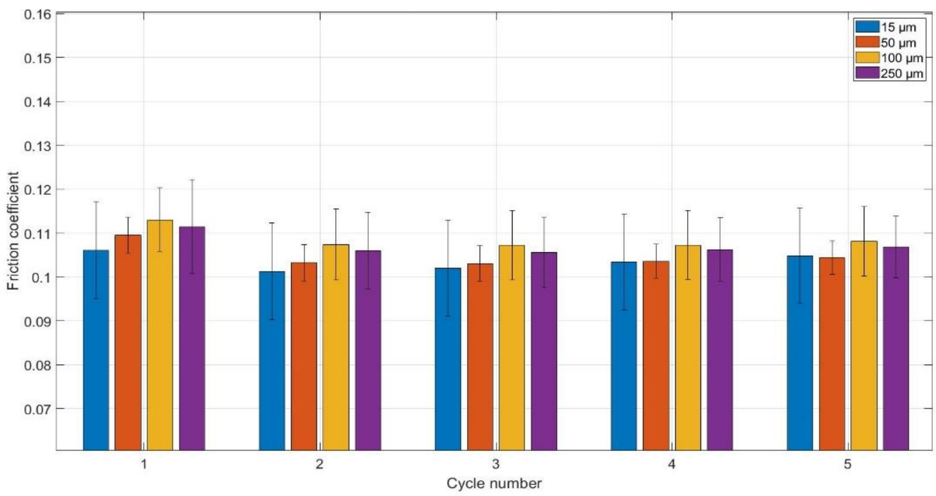
This comparison work will need to be completed in the future, in particular by seeking to relate the sensory data to the tongue deformation measurements that were made possible with the ultrasound measurements from this thesis work. However, we found that while these gel models were very relevant for validating the development of the ultrasound methods and their degree of sensitivity, they were not as relevant for the contrast of sensory perceptions reported by the panel.

Moving ahead, the ultrasound ToF was also used in chapter 4, for the first time, in an attempt to analyze the deformation of composite gels (bilayer) in relation to perception of heterogeneity. This work was inspired by the initial work of Santagiuliana et al. (2018) where on the same bilayer a sensory study gels was conducted. The protocol used in the said study did not impose any eating instructions, rather it allowed the panelists to freely consume the gels. The authors inferred from the obtained results that responsible factor in the perceptions of the heterogeneity was the difference in the fracture stress of the two layers. Our ultrasound method puts in evidence the mechanical aspect of the inference made by above mentioned article, as we quantified very efficiently the deformation undergone by each entity of the tongue-food system.

**<u>Sensory perceptions of heterogeneous foods v/s friction forces</u>**

Perception of heterogeneities in liquid food such as cottage cheese is however a complex phenomenon as it pertains largely to surface phenomenon or Tribology. Most of the work described in chapter 5b deals in understanding the how friction forces evolve when varying the properties of the biomimetic system (tongue properties, speed, and applied force). Again, it is interesting to look more closely at whether the information from the friction force analyses can be related to sensory perceptions for different food products. In the work presented in chapter 5b, the presence of heterogeneities in the cottage cheese (cellulose particles) was found to have a significant impact on the friction forces (almost more important than the rheology of the continuous phase).

Thus, the choice was made to conduct a study on the impact of the nature of the particles suspended in the cottage cheese: both on the friction forces measured on the experimental system, but also on the sensory perceptions during the consumption of the products. Two types of particles were considered: heterogeneously shaped cellulose particles (Vivapur) and spherical cellulose particles (Cellets, Pharmatrans Sanaq Ag, Basel-Allschwil Switzerland). Four different size ranges for each particle type was chosen; vivapur (15, 50, 100, 250 µm) and cellets (40-80, 80-125, 100-160 µm). The idea was to study the impact of the shape and size of the particles on the friction mechanisms and the sensory perceptions they generate. All this was done at particle size levels close to the human detection threshold. The 5% particles (w/w) were incorporated in the diluted cheese (50% w/w in water), similar to chapter 5b. The shearing was performed at 9 kPa normal stress and 10 mm/s shearing velocity. The obtained results are depicted in the figure 6.4. As already illustrated in Chapter 5a, the size of Vivapur particles was found to have little impact. It should be noted that they are heterogeneous in shape (see figure 6.5), which leads to significant uncertainty about their particle size, but also about their ability to move and influence lubrication when in contact with the asperities of the artificial tongue.




**<u>Figure 6.4:</u> Friction coefficient values for the diluted cottage cheese with vivapur particles on soft model tongue (50.17 ± 1.46 kPa) with rough surface profile. Normal stress and shearing velocity were 9 kPa and 10 mm/s, respectively. The error bars represent the standard deviations across six replicates.**

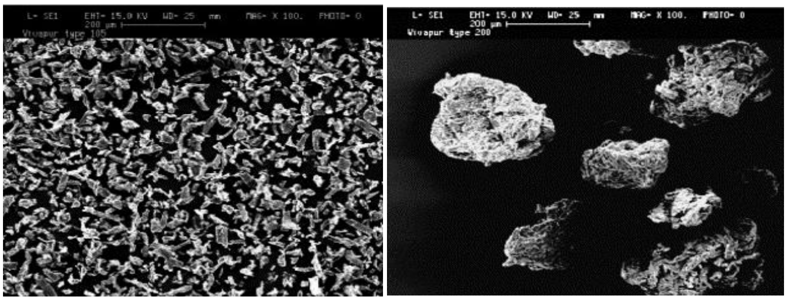



**Figure 6.5: Scanning electron microscopy of Vivapur samples at 100 x magnification. Left: Vivapur 105 (small particle used in this thesis, 15 µm); Right: Vivapur 200 (large particle used in this thesis, 250 µm); Di Martino et al. (2004).**

Conversely, the results obtained from hard, spherical particles (Cellets, see Figure 6.6) are encouraging in terms of the system's ability to highlight differences in friction phenomena depending on particle size. The smallest particles (present in greater numbers because we worked with the same mass concentration for the four references) were found to be associated with lower friction coefficient values. It can be seen in the figure 6.6 that these differences are maintained, cycle after cycle, throughout the test sequence. The spherical shape of the particles thus seems to be an important point.

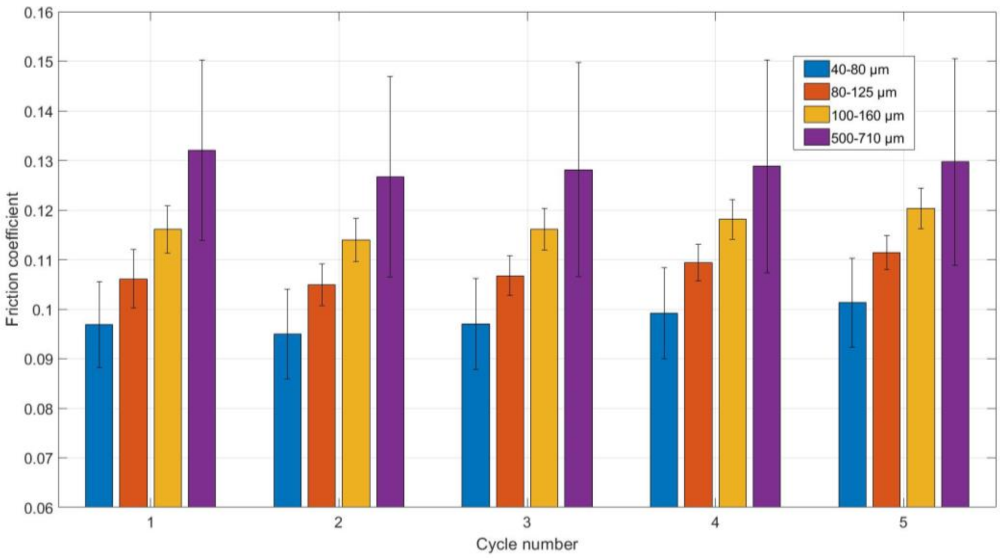
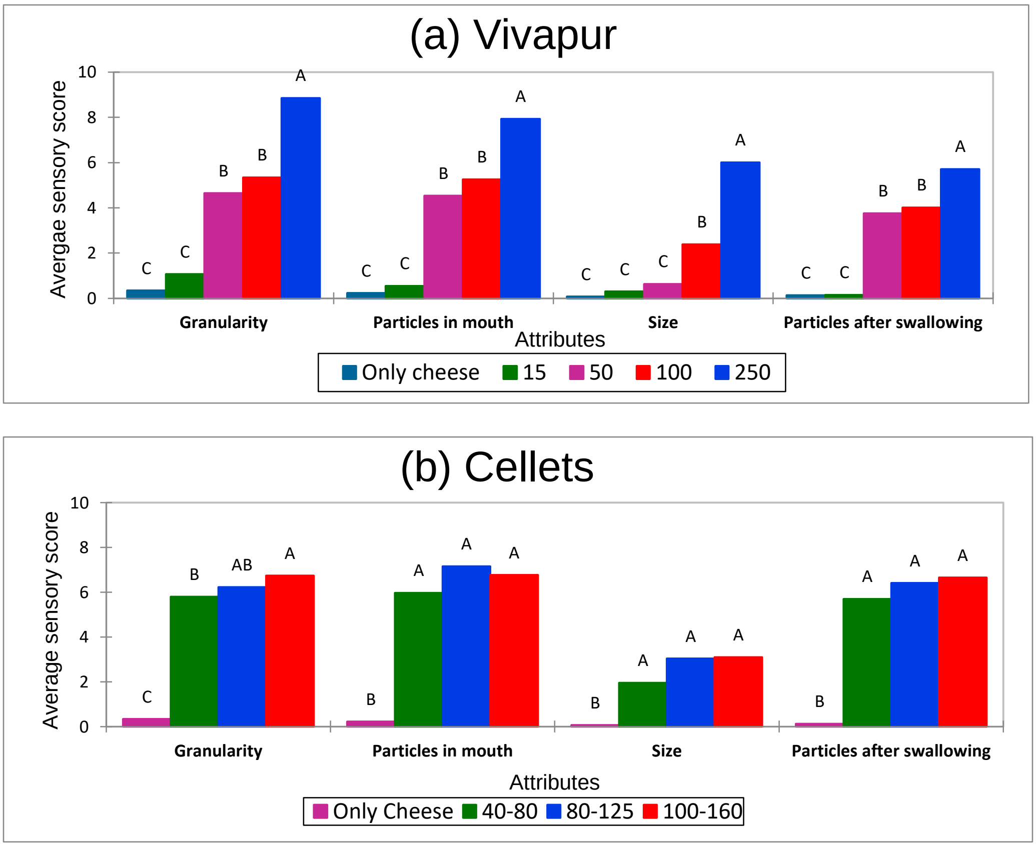



**Figure 6.6: Friction coefficient values for the diluted cottage cheese with cellets particles on soft model tongue (50.17 ± 1.46 kPa) with rough surface profile. Normal stress and shearing velocity were 9 kPa and 10 mm/s, respectively. The error bars represent the standard deviations across six replicates.**

A sensory study was performed (kindly refer to appendix) with a trained panel on cottage cheese incorporated with the two type of particles as mentioned above. The particles (5% w/w) were incorporated in undiluted cheese here (the dilution for tribology experiment was done to take into account the maximum extent of saliva incorporation). In figure 6.7a, it can be seen that panelists were able to perceive the differences in texture attributes for samples with vivapur particles of varied sizes (15, 50, 100, 250 µm). However, it was not the case (Fig 6.7b) for samples with cellets of varying size ranges (40-80, 80-125, 100-160 µm).




**<u>Figure 6.7:</u> Average sensory scores for the attributes (i) Granularity, (ii) Particles in mouth, (iii) Size and (iv) Particle after swallowing obtained for cottage cheese incorporated with (a) Vivapur (b) Cellets. The letters represent the statistical groups to which the averages belong statistical groups to which the means belong. Newman Keuls multiple comparison test, p<0.05%).**

Interestingly, where the friction measurements were able to differentiate spherical particles according to their size, it was not possible to do so as easily with the sensory panel. Conversely, the sensory panel was able to differentiate heterogeneously shaped particles very well, whereas the friction measurements did not. Thus, the work can question the relevance of the friction coefficient (or at least the conditions under which it was measured) in order to discriminate products according to the characteristics of the particles. Perhaps the ability to detect heterogeneities in the mouth with the tongue does not only involve shear movements. This assumption opens new prospects of combining shear and compression motions, along with monitoring of the tongue surface deformation with the ultrasound method.

# VII
# Conclusion & future prospects

The main objective of this thesis was to contribute to the need expressed by the Food Oral Processing community (i) to take into account the complexity of the physiological characteristics of the oral cavity during the manipulation of food between the tongue and the palate, and (ii) to pursue the development of instrumental methods allowing to follow the evolution of the mechanical behavior of foods (of increasing complexity) in interaction with the tongue and the palate. The combination of these two objectives should allow, in the long term, in support of sensory analysis, to better understand the mechanisms of texture perception and to propose new food offers that respond to societal challenges, while adapting to the specific needs of different types of populations.

Concerning the first part of the objective, we first relied on an experimental system considering only simple uniaxial compressions. The implementation of PVA gels as artificial tongues allowed us to demonstrate the particularly important role played by the roughness and deformability of the tongue, which are just as important as the properties of the food in understanding the mechanical response of the tongue-food-palate system. The design of a new biomimetic system, allowing to mimic both compressive and shear movements, has confirmed the interest and importance of the mechanical and topological properties of the tongue.

Concerning the second objective of the thesis, the work carried out has also brought significant advances. It has been possible to confirm the potential of ultrasonic methods for real-time monitoring (and non-destructively) of interactions between the tongue and the food: in particular by the coupling mechanisms at the interfaces, and by the ability to characterize the deformation rates undergone by the different components of the system. In addition, the work done to characterize the frictional response under shear has demonstrated the importance of the properties of the tongue, its speed of displacement, but also the normal force it applies on the palate, on the friction coefficient.

This work confirms that the characteristics of the tongue, until now little taken into account, can have an influence just as important as the type of food on the mechanical response obtained, whether in compression or shear. The oral physiological characteristics such as stiffness, roughness, speed of movement or applied force of the tongue are all properties that can vary significantly according to the wishes, preferences and abilities of individuals. And for the same individual, they can also vary during the course of food oral processing.

This thesis work opens up a large number of perspectives that could not be addressed during these three years. Among them, in the first place, one of the limitations that is easily opposed in the current approach is to have partly neglected the lubrication of the tongue by saliva. There is no doubt that saliva plays an essential role in lubricating the surface of the tongue, but also in participating in the mechanical breakdown of the food. The choice of PVA as a material for our artificial tongues should prove to be an advantage in this respect. Contrary to other polymers like PDMS, PVA gels have the advantage of offering appropriate wettability properties, allowing to consider the deposition of thin and stable lubricating films. A challenge will then arise: which saliva, or which artificial saliva, to use? Both solutions have their advantages and disadvantages and neither has really established itself in the literature.

Among the perspectives on the biomimicking system, it will also be a question of taking full advantage of the possibilities offered by the system by mimicking sequences of movements between the tongue and the palate that are more realistic. Ultrasound imaging could prove interesting in order to quantify movement sequences during food consumption by volunteers, and thus be able to implement them on the system. It would then be possible to make the link between the way a food is manipulated between the tongue and the palate, the physical mechanisms of interactions associated with it, and the resulting perceptions of texture. The development of biomimetic systems is of course not an end in itself. It is a means to better understand the phenomena that occur in the oral cavity, by mastering the complexity of each of the components, and by combining instrumental measurements that are not always easy to use in *in vivo* conditions. The perspectives of this thesis work will therefore logically include the further development of instrumental methods, and their implementation to understand the biomechanics of the tongue-palate system.

The results obtained with the new device show that the friction forces depend strongly on the conditions inherent to the tongue and its environment, perhaps even more on these conditions than on the properties of the food. Moreover, the results also suggest that the friction forces measured here cannot alone explain the entire tactile dimension of texture perceptions. For example, we could see that friction forces could be sometimes less efficient, and in other cases more efficient than sensory characterizations in order to distinguish the nature of the particles present in cottage cheese (according to their shape, their size, their hardness). Thus, it is essential to continue the development of friction measurement methods under realistic conditions, but keeping in mind that they are not sufficient to understand the full tactile dimension of texture perceptions.

An aspect still often neglected by the food oral processing community is the frequency range of sensitivity of mechanoreceptors. If some are said to be slowly adapting, others, which are rapidly adapting, are more sensitive to the onset and cessation phases of stimuli, with frequencies that can reach several hundred Hertz. It is therefore of great interest to measure the vibratory response during the manipulation of the food between the tongue and the palate. This is something that can be done with the accelerometer installed on the system.

Finally, the use of quantitative ultrasound methods will be central to the logical continuation of the work. Because of their non-invasive and non-destructive character, they have a real potential to carry out in vivo measurements, directly on the consumer. While waiting to go that far, new approaches can be considered on the in vitro systems developed. The work done during the thesis showed a great potential to follow the couplings at the interfaces, but the use of single-element transducers implied to study heterogeneities oriented perpendicularly to the beam axis. The use of multi-element line probes, like those found in imaging (both straight and convex), could also be considered. It would thus be possible to study more easily the media whose heterogeneities are not only in the axis of the ultrasonic beam, but also in the width of the probe. The progression towards more and more refined methods will also allow to go further, in particular with the use of faster systems (with recurrence frequencies of ultrasonic shots of the order of kHz) to consider following the vibratory events mentioned above.

*To conclude, this PhD project was carried out with the aim to contribute towards biomechanical characterization of the tongue-food interface through different novel in vitro approaches and has presented exciting observations which will make us advance in understanding the phenomena behind texture perception. The work also opens up exiting challenges for food oral processing community in terms of bridging further the gap between real oral physiology and instrumental methods especially on key aspects like tongue roughness, stiffness and wettability. These different perspectives will thus allow to lift the technical and scientific barriers to study the mechanisms responsible for the perception of texture directly on the consumer.*

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# Appendix

**Sensory analysis method**

The "descriptive profile" or QDA method was used. It consisted of recruiting a panel, capable of generating a list of sensory attributes allowing to describe and discriminate the products to be tested. The panel was trained to describe the attributes and their intensity on a defined scale in order to be able to evaluate the products within the study. The study period lasted seven weeks.

**Recruitment**

A panel of 10 volunteers (7 females and 3 males) ranging in age from 21 to 58 years was recruited for this study. They were in good health and had no food allergies. They were informed about the general context of the study and the conditions of evaluation, and gave their free consent to participate in this study.

**Training**

A total of five (30 min) training sessions were conducted. The first two sessions were used for the generation, definition, and selection of texture descriptors, as well as the development of the operating procedure for each descriptor. In addition, the second session allowed the panel members to become familiar with the recognition of the different terms by proposing training products for tasting that were representative of each descriptor.

This work was carried out on different products procured directly from the market (such as cream cheese (Philadelphia, Mondelez International, France), and cottage cheeses with 0% and 7.8% fat content (Fromage blanc, Auchan, France), as well as on products formulated in the laboratory from commercial products (cottage cheeses mixed with different concentrations of poppy seeds and couscous semolina of fine and medium size). These reference foods were studied because they present a wide spectrum of texture perceptions; the instructions given to the judges were to focus only on texture properties, independently of potential flavor and aroma perceptions. Following these first two sessions, the relevant descriptors having been defined and understood, the panelists were trained to use the FIZZ software (Version 2.61.0, Biosystemes, Couternon, France). This software allows the design of tests in a digital way, the automated collection of data, their analysis, as well as their management.

The next two training sessions focused on the evaluation of the intensity of the descriptors on scales of intensity ranging from "not very intense" to "very intense". For this, each judge was given three products per attribute to rank on the scale. The foods tested were similar to those evaluated during the final phase of the study: 0% fat cottage cheese was mixed with vivapur and cellets particles in different concentrations. Finally, the last session allowed the evaluation of three products in replicates under real examination conditions in order to verify and validate the panel's findings. The final choice of attributes were:

| **Attributes** | | **Scale** | **Definitions** | **Protocol** |
|---|---|---|---|---|
| Overall flavor intensity | **« Taste »** | from "no taste" to "intense" taste | Note if the product has a particular taste so that you can focus only on texture for other attributes. | Place a spoonful of product in the center of the tongue and circulate it in the mouth. |
| *Overall product (granularity))* | **« Granular »** | from "smooth" to "granular" | Evaluation of the criteria smooth to granular, due to the presence or not of particles within the product. | Place a spoonful of product in the center of the tongue and circulate it in the mouth. |
| *For continuous phase (consistency)* | **« Pasty »** | from "slightly pasty" to "very pasty" | Evaluation of the resistance of the product to compression between the tongue and palate. | Place a spoonful of product on the tongue and squeeze it at a slow speed between the tongue and the palate. Measure the resistance of the product. |
| | **« Mouth coating »** | From low to high | Evaluation of the palate covering by the product. | Place a spoonful of product in the center of the tongue and circulate it in the mouth; then swallow. Evaluate the quantity of product covering the palate (film that lines it). |
| *Specific to heterogeneities* | **«Number in mouth »** | from "few particles" to "many particles" | Evaluation of the number of particles between the tongue and the palate. | Place a spoonful of product in the center of the tongue and crush it on the palate while sweeping with the tongue. |
| | **« Size »** | from "small particles" to "large particles" | Evaluation of the size of the particles between the tongue and the palate. | Place a spoonful of product in the center of the tongue and press it to the palate while sweeping with the tongue. |
| | **« Hardness »** | from "soft particles" to "hard particles" | Evaluation of the hardness of the particles between | Place a spoonful of product in the center of the tongue and press it to the palate |

| | | | | |
|---|---|---|---|---|
| | | | the tongue and the palate. | while sweeping with the tongue. |
| Oral processing | **« Number after swallowing »** | from "few particles" to "many particles" | Evaluation of the number of particles remaining in the mouth after swallowing. | Place a spoonful of product in the center of the tongue and circulate it in the mouth; then swallow. |

**Product**

The cottage cheese (Fromage blanc, Callin 0% extra, Yoplait, France) was used the carrier medium (undiluted) for following particles:

1. Vivapur are hard microcrystalline cellulose particles with no uniform shape. The particle size represented below is the average size provided by the supplier.

| | **VIVAPUR® 105** | **VIVAPUR® 101** | **VIVAPUR® 102** | **VIVAPUR® 200** |
|---|---|---|---|---|
| *Diameter (µm)* | 15 | 50 | 100 | 250 |

2. Cellets are also hard microcrystalline cellulose particles with uniform spherical shape. . The particle size represented below is the range provided by the supplier.

| | **CELLETS® 90 Sieved** | | **CELLETS® 127** | **CELLETS® 500** |
|---|---|---|---|---|
| *Diameter (µm)* | 40-80 | 80-125 | 100-160 | 500-710 |

These particles were incorporated into the cheese preparations with a concentration level of 1 and 5%.

*Quality check: Microbiological assessment by an external laboratory (Eurofins, Microbiologie Ouest, Nantes, France) was carried out for each product and allowed to validate their safety for consumption by the panel. Each sample prepared and analyzed in microbiology consisted of 95g of cottage cheese mixed with 5g of particles. During the preparation of the products containing the deformable micro-gel particles, the pH of each solution containing the particles was measured to verify that its value was between 4.2 and 4.5.*

**Evaluation**

All sensory analysis sessions took place in a sensory evaluation room, air-conditioned at 20°C and ventilated, consisting of individual tasting booths, each equipped with a computer to record the judges' responses, and under white light. The answers were collected by the FIZZ software.

The evaluation sessions lasted for 30 min and allowed the products to be evaluated three times for each product, two five-gram spoons were made available to the judges. The products were served at 20°C and presented simultaneously (each product was present in front of the panelist at the beginning of the session). The tasting was done according to an established order by Williams design method (different order from one judge to another, to balance the effect of order). Each sample was coded with three random numbers. Judges used a structured rating scale with bounds ranging from "not very intense" to "very intense" to rate the intensity of descriptors. Panelists were provided with the spoons containing the product, as well as a bottle of water for mouthwash and unsalted cookies.

**Statistical tool**

The statistical analyses of the sensory data were performed with the XLSTAT software. Decision statistics were performed using the analysis of variance (ANOVA) to determine for a given variable if there were any significant differences.

**ECOLE DOCTORALE N°581**
Agriculture, alimentation, biologie, environnement et santé (ABIES)

**Titre :** Caractérisation biomécanique de l'interface langue-aliment au cours du processus oral

**Mots clés :** Biomécanique, Perception de la texture, Ultrasons, Structures des aliments, Analyse sensorielle

**Résumé :** La transformation orale des aliments et la biomécanique associée jouent un rôle important dans la dynamique de la perception de la texture. Cette thèse avait pour but de comprendre cette biomécanique en concevant de nouveaux outils d'étude in vitro du processus oral. Des modèles de langues ont été fabriqués à partir d'alcool polyvinylique (PVA), ce qui a permis d'imiter les caractéristiques de la langue humaine comme la rigidité et la rugosité de surface. En outre, de nouvelles techniques ultrasonores ont été développées pour l'analyse en temps réel du système langue-aliment-palais. Dans un premier temps, la déformation de gels de gélatine et d'agar sur les modèles de langues a été étudiée lors de compressions uniaxiales. Deux paramètres ultrasonores (coefficient de réflexion apparent et temps de vol) ont été identifiés comme pertinents pour l'analyse (i) du couplage entre la langue et l'interface alimentaire et (ii) des déformations de la langue. Les résultats ont montré que les propriétés de la langue (rigidité, rugosité) et celles de l'aliment (rigidité et synérèse) jouaient toutes deux un rôle capital. La méthode ultrasonore a ensuite été développée pour analyser les déformations au sein de gels composites multicouches. Pour la première fois, il a été possible d'observer en temps réel la déformation de chaque couche de gel au sein de l'aliment et d'apporter ainsi des connaissances nouvelles pour mieux comprendre les perceptions d'hétérogénéité de textures.

Pour aller plus loin, un nouveau dispositif permettant d'effectuer des séquences de mouvements entre langue et palais plus complexes (compression et cisaillement) a été mis au point. Il a été équipé de capteurs de force à trois axes pour évaluer les forces de friction. La faisabilité du prototype a été testée en étudiant comment les valeurs du coefficient de friction étaient affectées par les propriétés de l'aliment (fromage blanc : principalement sa viscosité ainsi que la présence de particules), les propriétés de la langue et les paramètres opérationnels (contrainte normale, vitesse de cisaillement). L'augmentation de la rugosité de la surface et de la rigidité de la langue modèle a entraîné une augmentation prononcée des valeurs du coefficient de friction. Une analyse sensorielle a également été réalisée pour mettre en relation les phénomènes mécaniques observés grâce aux systèmes expérimentaux mis en oeuvre. Les résultats confirment qu'au-delà des caractéristiques physico-chimiques des aliments, il est essentiel de prendre en compte les caractéristiques de la langue (et sa variabilité) afin de mieux comprendre les interactions mécaniques qui affectent les perceptions de textures.

**Title :** Biomechanical characterization of the tongue-food interface during the course of oral processing

**Keywords :** Biomechanics, Texture perception, Ultrasound, Food structure, Sensory analysis

**Abstract :** Food oral processing and associated biomechanics play important role in the dynamics of texture perceptions. This thesis was aimed to understand this biomechanics by designing novel in-vitro oral processing setups. Model tongues were fabricated from polyvinyl alcohol (PVA), which helped to mimic human tongue characteristics like rigidity and surface roughness. Furthermore, novel ultrasound techniques were developed for real-time analysis of the tongue-food-palate system. At first, the deformation of gelatin and agar gels over the model tongues were investigated during uniaxial compressions. Two ultrasound parameters (apparent reflection coefficient and time of flight) were identified as suitable for analyzing (i) tongue-food interface coupling and (ii) tongue deformations. The results showed that both tongue properties (rigidity, roughness) and food properties (rigidity and syneresis) were critical. The ultrasound method was then further developed to analyze bulk deformations of composite gels in context of perception of heterogeneity. For the first time, it was possible to observe in real time the deformation of each gel layer within the food and thus provide new knowledge to better understand the perceptions of texture heterogeneity.

Moving forward, a new set-up that could perform more complex motion sequences (compression and shearing) was developed. It was equipped with three-axis force sensors to evaluate friction forces. The feasibility of the set-up was tested by measuring how friction coefficient values were affected by food properties (cottage cheese: mainly its viscosity and the presence of particules), tongue properties, and operational parameters (normal stress, shearing velocity). Increases in the surface roughness and bulk rigidity of the model tongue led to a pronounced augmentation of the friction coefficient values. Sensory analysis was also carried out to relate the mechanical phenomena observed with the two set-ups. The results confirm that beyond the physico-chemical characteristics of food, it is essential to take into account the tongue characteristics (and variability) in order to better understand the mechanical interactions which likely affect texture perceptions.

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