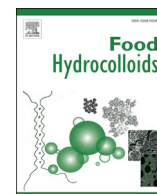


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Characterisation of friction behaviour of intact soft solid foods and food boli

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A B S T R A C T

Methodologies to quantify friction forces between soft solid foods or food boli and (model) oral surfaces are desired to better understand how changes in food properties during oral processing affect sensory perception. In this short communication, friction forces (F_F) occurring between polydimethylsiloxane (PDMS) surfaces and intact soft solid foods/boli were quantified. As models for intact foods, we used gelatine gels varying in composition and particle size, and sausages were used as an example for real foods. Friction forces measured during the relative motion of intact foods against a rough PDMS surface ("oral surface"), strongly depended on the composition of the food. Friction forces were significantly lower for PDMS against emulsion-filled gels, than for PDMS against unfilled gels, likely due to the lubricating effect of released oil from the gel. Moreover, sausages, displayed significantly higher friction forces than gelatine gels when moving against the PDMS probe, presumably linked to differences in the surface of the foods. The friction forces observed for the PDMS probe moving against food boli were dependent on particle size and saliva quantity; boli with larger particle sizes showed significantly lower friction forces, whereas the addition of saliva to food boli first increased friction forces, but with increasing amount decreased the friction forces significantly. We conclude that the presented methodology is able to quantify the friction behaviour of intact soft solid foods and boli directly, taking into account (i) the effect of composition and added fillers, (ii) serum or oil release and (iii) bolus particle size.

1. Introduction

The structure of soft solid foods changes dynamically throughout its consumption; food is reduced in size, mixed with saliva and enzymes, and a cohesive mass is formed (Aguayo-Mendoza et al., 2019; Stieger & van de Velde, 2013). During this process, the mechanical properties of food change, as does the perception (Chen, 2009; Young, Cheong, Hedderley, Morgenstern, & James, 2013). In order to know how processing and composition of foods affect sensory perception, the dynamic changes in the rheological and tribological behaviour of foods have to be studied (Dickinson, 2018; Stokes, Boehm, & Baier, 2013). The relationships between lubrication properties and sensory perception have already been discussed for many liquid foods, including milk or o/w emulsions (Carvalho-da-Silva, Van Damme, Taylor, Hort, & Wolf, 2013; Dresselhuis et al., 2007; Laguna, Farrell, Bryant, Morina, & Sarkar, 2017; Sarkar, Andablo-Reyes, Bryant, Dowson, & Neville, 2019; Wang & Chen, 2017), and gel-like foods with irreversible spreading properties, such as custards (de Wijk, Prinz, & Janssen, 2006), mayonnaises

(Douaire, Stephenson, & Norton, 2014), and fluid chocolate samples (Carvalho-da-Silva et al., 2013). The lubrication behaviour of such foods are often assessed in tribological setups in which the liquid food lubricates the contact area between a flat surface and a spherical probe. The surfaces are typically made of polydimethylsiloxane (PDMS), glass, steel or animal tissue (Chen, 2014; Efimenko, Wallace, & Genzer, 2002; Ranc, Servais, Chauvy, Debaud, & Mischler, 2006; Selway & Stokes, 2013; Dresselhuis, de Hoog, Cohen Stuart, Vingerhoeds, & van Aken, 2008). In contrast to liquid foods, less is known about the tribological behaviour of soft solid foods moving against oral surfaces; specifically, there is a lack in approaches for the characterisation of the tribological behaviour of soft solid foods. In this case, we refer to soft solid foods as self-supporting gel-like foods with reversible behaviour (Dickinson, 2013; Vliet, 2013), such as cheeses and sausages. Some studies have been performed on soft solid foods that were reduced in size to obtain suspensions of particles to entrain those particles between two solid surfaces as a lubricant. Krop and co-workers, for example, determined lubrication properties of soft solid food boli gel particles using a PDMS ball and PDMS disk setup

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(Krop, Hetherington, Holmes, Miquel, & Sarkar, 2019). Liu and co-workers quantified the tribological behaviour of soft solid broken down gel particles entrained between PDMS and glass surfaces (Liu, Stieger, van der Linden, & van de Velde, 2016). To the best of our knowledge, tribological characterisation of intact soft solid foods in a mouth-mimicking setup, has not been reported. However, extensive research has been performed on hydrogels prepared from non-food grade materials, such as polyvinyl alcohol, poly (2-acrylamido-2-methylpropanesulfonic acid) or poly(acrylic acid) (Gong, 2006; Gong & Osada, 2002; Oogaki et al., 2009; Zhao, Liu, & Liu, 2018). From studies on hydrogels, we know that the friction behaviour can deviate from that of solid materials (Gong, 2006; Oogaki et al., 2009; Rudge, Scholten, & Dijkstra, 2019). Important parameters that have been shown to affect the friction behaviour include charge (Liu, Thormann, Tyröde, & Claesson, 2015), porosity (Caravia, Dowson, Fisher, Corkhill, & Tighe, 1993), substrate effects (Gong, 2006; Gong & Osada, 2002) and the presence of polymer brushes (Ishikawa, Hiratsuka, & Sasada, 2006). Furthermore, adhesion or repulsion between the probe and the gel can strongly interfere (Gong, 2006). Although many factors in hydrogels have been studied, intact food gels are largely unexplored. The difference between polymer gels and soft solid foods lies in the high compositional complexity and heterogeneity of soft solid foods. Foods consist of mixtures of proteins, polysaccharides and oil, which often have ill-defined properties and are polydisperse in nature. It would be beneficial to understand the friction behaviour of intact soft solid foods in contact with surfaces simulating oral surfaces, but also food boli at different stages of mastication (Pradal & Stokes, 2016). This can contribute to understanding sensory perception during different stages of oral processing, such as sticky, fatty and mouth coating sensations. This study, therefore, aimed to find an approach allowing to quantify friction forces (F_f) occurring between intact solid foods and food boli and simulating oral surfaces. We specifically test whether small changes in composition can be quantified by taking into account (i) addition of different dispersed particles to the gel matrix, (ii) expelled serum or oil from the matrix and (iii) effect of bolus particle size. We also incorporate saliva into the food (bolus), as this is well known to increase the oral lubricity of foods. We propose an experimental approach using a flat, cylindrical PDMS surface to mimic the oral surfaces, which is in direct contact with the intact soft solid food. For comparison, we examined intact and broken-down sausages, simulating real food boli at different stages of oral processing.

2. Materials and methods

2.1. Materials

Polydimethylsiloxane (PDMS 184 silicone elastomer kit) was bought from Dow (Dow silicones, Dow Europe GmbH, Wiesbaden, Germany). Ethanol was purchased from Sigma-Aldrich (Steinheim, Germany). Hot dog sausages (Unox, Unilever, Rotterdam, Netherlands) were kindly provided by Unilever Nederland BV. Potato starch (Honig, Amsterdam, Netherlands) and sunflower oil (Reddy, Vandermoortele, Gent, Belgium) were purchased at a local supermarket. Gelatine (from pork skin, bloom 250, isoelectric point of 8–9) was obtained from Rousselot (Gent, Belgium). Unstimulated saliva was collected as described in literature (Silletti, Vingerhoeds, Norde, & van Aken, 2007). In short, the saliva of one, generally healthy volunteer was collected, in the morning. The participant was asked to abstain from food intake for 1 h before collection. Before the saliva collection, the participant was asked to rinse the mouth with water and collect saliva for 1 min; this saliva was discarded. Subsequently, unstimulated saliva was collected. The saliva was centrifuged at 4 °C at 10000 g for 30 min to remove debris (Beckmann, Avanti TM J-25 I, JA-21, Beckman Coulter B.V. Mijdrecht, The Netherlands) and kept on ice. Saliva was then used with no further storage. Demineralised water was used for all experiments unless stated otherwise.

2.2. Preparation of model soft solid foods and *in vitro* masticated boli

Three model soft solid foods differing in composition were prepared. The samples consisted of gels with gelatine as the continuous protein matrix and included oil droplets or starch granules as the dispersed phase. The composition, mechanical properties of the model systems and an overview of all samples used for tribological tests are shown in Table 1.

Gelatine (12.5%) was dispersed and hydrated in cold water for 2 h, and subsequently melted in a water bath at 80 °C for 30 min. The pH of the gelatine dispersions was 7. Thus, the gelatine was slightly positively charged. To obtain starch-filled gelatine gels, gelatine solutions were mixed with a potato starch suspension (12.5% (w/w)). The mixture was blended using a high-speed blender for 3 min at 10000 rpm (Digital Ultra Turrax T25, IKA Werke, Germany). The final mixture contained 7.5% (w/w) gelatine and 5% (w/w) starch. To obtain emulsion-filled gelatine gels, liquid solutions of gelatine (12.5%) were first diluted with water, and then mixed with sunflower oil to reach a final concentration of 14% (w/w) oil and 7.5% gelatin. The blend was mixed with a high-speed blender for 1 min at 10000 rpm and homogenised in three cycles at 180 bar (LabhoScope homogeniser, Delta Instruments, Drachten, Netherlands). Gelatine gels without additional components were obtained by diluting gelatine solutions with water to a final protein content of 7.5% (w/w). All warm, liquid mixtures (80 °C) were poured in Petri dishes (diameter 85 mm) to a height of 7 mm and stored at 4 °C overnight to solidify. After solidification, gels were cut into cubes of 55 × 40 × 7 mm. Commercial hot dog sausages were cut along their longitudinal plane with a scalpel into cylinders of 55 mm length and 20 mm diameter.

To obtain “*in vitro* masticated” food boli, gels and sausages were ground using a meat grinder (Westmark, Lennestadt, Germany). The particle size of the boli was varied using two different pore sizes (4.5 and 8.0 mm) of the grinder to mimic boli during different stages of oral processing. Various concentrations of saliva (5, 10, 20, 30 and 40% (w/w)) were added to the boli. Saliva (unstimulated) was collected as described in the materials section 2.1. Saliva was incorporated by mixing 12 g of ground boli and the respective amount of saliva in a 60 mL syringe (BD Plastipak Luer-Lok, USA) and careful pumping the plunger 20 times; no considerable changes in particle size (4 ± 2 mm and 7 ± 3 mm) were observed. This methodology is based on the procedure described by Ishihara, Nakauma, Funami, Odake, and Nishinari (2011).

2.3. Mechanical properties of soft solid food gels and sausages

Cylindrical pieces of gels (27 × 20 mm) and sausages (20 × 20 mm) were analysed in a Texture Analyser (TA XT plus Texture Analyser, Texture Technologies, Hamilton, USA). Gels were compressed at a rate of 1 mm/s with an acrylic disk (diameter 100 mm) until 80% strain was reached. Surfaces were wetted with paraffin oil to avoid friction. Measurements were performed at room temperature (24 ± 2 °C) in triplicate. Young's Modulus, fracture stress and fracture strain were obtained from the stress-strain curves and averaged over replicates.

2.4. Methodology to measure tribological behaviour of soft solid gels and food boli

A schematic representation of the measuring setup can be found in Fig. 1. Friction behaviour was measured with a commercially available universal mechanical tester “Tribolab” (UMT, Bruker, Billerica USA). As a lower drive, a reciprocating drive (5 mm stroke length, reciprocating frequency ranging from 0.01 to 8.25 Hz) was used in combination with a holder, lined with a smooth PDMS surface (60 × 40 × 4.5 mm), facilitating the fixation of the sample in the holder (Fig. 1). Intact soft solid gels were mounted in the holder and fixed with metal plates and screws to avoid slip or buckling. Thus, the tribological pair for the intact soft solid foods is PDMS/gel (Fig. 1c), where the PDMS simulates oral

Table 1
Overview of all intact soft solid foods and model food boli used for the tribological measurements.

Sample	Composition			Mechanical properties			Intact soft solid food	Soft solid food bolus ^c
	Protein (% w/w)	Fat (% w/w)	Starch (% w/w)	True fracture stress (kPa)	True fracture strain (-, ×100 for %)	Young's modulus (kPa)	Lubricant (on gel) ^a	Lubricant (incorporated) ^b
Sausage	14	14	5	33.1 ± 0.5	0.56 ± 0.03	33 ± 8	water, saliva, oil	saliva
Gelatine gel	7.5	0	0	34.0 ± 2.0	1.31 ± 0.03	8 ± 3		
Starch-filled gelatine gel	7.5	0	5	20.5 ± 2.9	1.12 ± 0.07	4 ± 1		
Emulsion-filled gelatine gel	7.5	14	0	40.5 ± 1.2	1.13 ± 0.05	18 ± 1		

^a Volume of lubricant added on top of intact soft solid food was 2.5 mL.

^b Concentration of saliva as a lubricant added into bolus was varied (5, 10, 20, 30 and 40% (w/w)).

^c Particle size of broken down soft solid food boli were 4.5 and 8.0 mm.

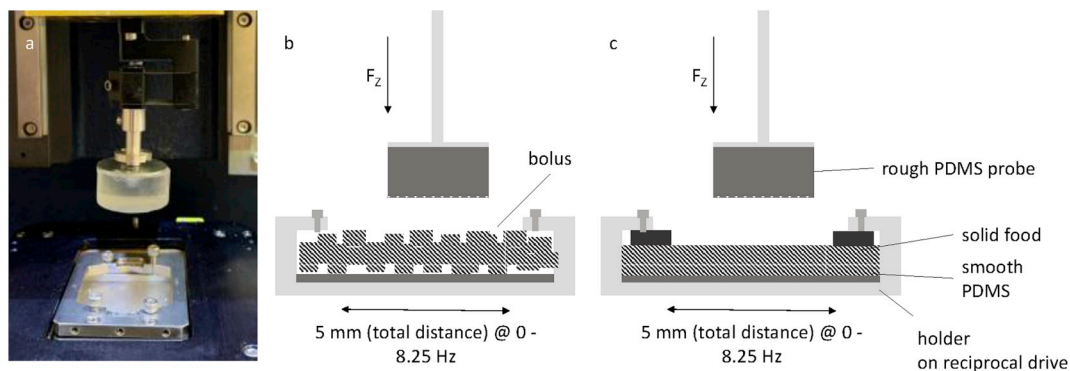


Fig. 1. Overview of the tribological setup. a: Measurement setup with intact soft solid food gel, b: schematic representation of tribological setup with broken down food bolus particles (height bolus/gel 7 mm), c: schematic representation of the tribological set up with soft solid food.

surfaces. When the intact gel was used, the friction force was determined by the contact of the flat gel surface area with the upper probe. Broken down fragments were placed into the liquid holder without further fixation. Sufficient bolus material was added to ensure sufficient contact between the bolus material and the probe during the measurement. Thus, the tribological setup for the food boli is PDMS/food bolus particles (Fig. 1b). One has to consider in this setup that two friction forces are involved, (a) the friction force between probe and bolus sample and (b) between bolus particles within the bolus.

Samples, having a height of 7 mm, were brought into contact with the upper probe (rough PDMS) with a normal force of 0.5 N. A cylindrical upper probe, with curved edges, made of roughened PDMS (30 × 15 mm, 706 mm²) was designed in-house. PDMS was mixed with the supplied crosslinker in a 10 to 1 vol ratio, as reported previously (Dresselhuis, de Hoog, Cohen Stuart, & van Aken, 2008) and subsequently de-aired for 2 h at room temperature. A mould for the cylindrical upper probe was coated with sandpaper (size 240, corresponding to an average particle diameter of 53 μm, according to ISO 6344 (1998)) to create a rough PDMS surface. Following de-airing, the sample was cured at 60 °C overnight. The modulus of the PDMS probe is about 2 MPa. A 20 N friction/load sensor (DFM-2G, Bruker, Billerica USA) was used.

The friction force (F_F) was subsequently measured at speeds ranging from 0.1 mm/s to 90 mm/s. Measurements were performed in triplicates, and average values with standard deviation obtained. Measurements were analysed for significant differences using a two-way ANOVA and a Bonferroni post-test with $p < 0.05$. We compared means at one speed of different samples as well as differences within one sample due to differences in sliding speed.

Additionally, intact soft solid foods were measured in the presence of lubricants (water, saliva, oil) added on top of the surface of the soft solid food. Lubricants (2.5 mL) were added with a pipet directly before the measurement onto the surface of the intact gel. By those means, the

effect of the presence of lubricants on the surface of soft solid foods on friction behaviour was assessed. This was done to mimic the release of liquid from the food during mechanical testing and to mimic saliva incorporation into the food bolus during oral processing.

3. Results and discussion

The mechanical properties of the gels and sausage are given in Table 1. As can be seen, the addition of starch reduced the fracture stress, fracture strain and Young's modulus of gelatine gels, while the incorporation of oil droplets increased fracture stress and Young's modulus and decreased fracture strain. The friction force (F_F), measured between the intact soft solid foods and the mouth-mimicking PDMS, is given as a function of speed in Fig. 2. Friction force (F_F) increased with increasing velocity, even to values higher than the applied normal force (0.5N). We suggest that this is due to adhesive forces and possibly attractive electrostatic interactions between positively-charged gelatine and negatively-charged PDMS (Beal, Bubendorfer, Kemmitt, Hoek, & Mike Arnold, 2012; Gong, 2006). The friction force (F_F) increased until a sample-dependent plateau value of around 0.6–0.8 N was reached. Over the entire speed range, differences in friction behaviour between gels can be observed. The friction forces (F_F) recorded for the intact gels are related to the composition of the gels (Fig. 2a). Low friction forces were obtained for emulsion-filled gelatine gels, with significant differences ($p < 0.05$) between the non-filled gel and the emulsion-filled gel in the range from 1 to 10 mm/s. Starch-filled gelatine gels displayed a similar friction force as the non-filled gels (differences are not significant). The lower F_F for oil-containing gels could be related to either oil release or serum release. We suggest that oil release is mostly responsible for this observation, as after the measurements, small patches of oil were observed on the surface of the emulsion-filled gelatine gel. The oil droplets are probably expelled during friction measurements, which is

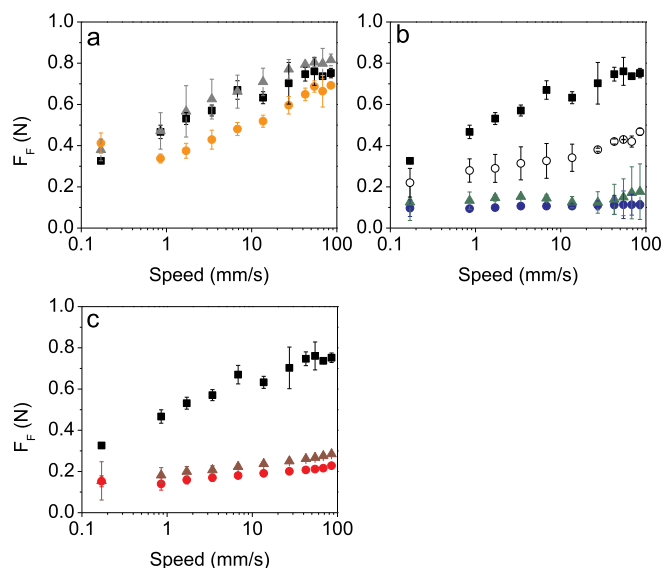


Fig. 2. Friction force (F_F) between soft solid gelatine gels against PDMS probe, as a function of speed. a: Gelatine gels differing in composition/filler, without added lubricant. Black squares: gelatine gel, orange circles: emulsion-filled gelatine gel; grey triangles: starch-filled gelatine gel. ($F_{\text{sample}}:14.94$, $p < 0.001$; $F_{\text{speed}}: 72.48$, $p < 0.001$) b: Gelatine gel, without filler with various added lubricants (on surface). Black squares: no lubricant, blue circles: water, green triangles: saliva, open circles: oil. ($F_{\text{sample}}:83.81$, $p < 0.001$; $F_{\text{speed}}: 4.28$, $p < 0.001$) c: Gelatine gel boli. Black squares: (intact) gelatine gels. Red circles: 8 mm particles; dark red triangles: 4.5 mm particles. ($F_{\text{sample}}:83.97$, $p < 0.001$; $F_{\text{speed}}: 9.34$, $p < 0.001$) The average from 3 measurements is shown with error bars representing a 95% confidence interval. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

supported by literature, in which it was shown that gelatine gels can release oil droplets from emulsion-filled gels due to shear processes (Sala et al., 2007). As gelatine has a very high water holding capacity, serum release is less likely (Martin et al., 2016). Although not significant, gels containing starch showed a slight trend for higher friction forces (F_F) than unfilled gels. This may be due to the starch granules providing a more rough or sticky surface, or the additional binding of free water by starch granules. Our setup is, thus, capable of distinguishing friction behaviour of gels differing in composition. Besides the properties of the continuous phase, also the dispersed phase affects the friction behaviour.

Water, human saliva, and oil were individually added as lubricants on the surface of the gelatine gels without fillers to investigate the effect of liquids on the gel surface on friction behaviour (Table 1). As expected, the friction force (F_F) decreased significantly ($p < 0.05$) upon addition of water, saliva and oil (Fig. 2b), indicating that all tested liquids lubricated the surface of the gelatine gels to some degree: samples lubricated with water or saliva showed the highest decrease, to 0.1 N, but showed no significant difference ($p > 0.05$) between each other. The addition of the lubricants decreased the adhesive forces. Oil decreased friction slightly less than the aqueous lubricants to a friction force of 0.2–0.4 N ($p < 0.05$). The reason for the observed differences in friction behaviour lies in the surface wettability of the gel. Gelatine is a highly hydrophilic matrix, and therefore has high affinity for hydrophilic lubricants, such as water and saliva. Hydrophobic oil cannot wet the surface as efficiently as the water-based lubricants, thus friction forces are higher. When these lubricants were added on top of the surface of emulsion-filled gelatine gels or starch-filled gelatine gels, similar trends were obtained (data not shown).

Upon producing an artificial bolus and changing the contact type of the tribological setup from a PDMS/gel to a PDMS/bolus contact

(Fig. 2c), a substantial decrease in friction force is observed. However, the observed friction force did not differ significantly ($p > 0.05$) between model boli with a particle size of 4.5 and 8 mm. As the food is broken down into smaller bolus particles, the apparent contact area between the bolus particles and the upper probe decreases, as the probe is now not in contact anymore with the sample over the entire area. Therefore, adhesion between the probe and the bolus particles is reduced, as compared to the intact gel and thus the friction force was observed to decrease ($p < 0.05$). It should also be taken into account that the bolus has to be considered as a deformable viscoelastic material. The friction force is therefore not only depended on the surface properties, but is also influenced by particle-particle interactions, deformation of the gel particles, and energy dissipation during movement (Rigney & Hirth, 1979).

The friction forces between the rough PDMS and (i) the intact sausages and (ii) simulated sausage boli, varying in bolus particle size (4.5 mm, Fig. 3a; 8 mm, Fig. 3b) and amount of lubricant (saliva) are shown in Fig. 3. Intact solid sausages with added saliva led to overall higher friction forces (F_F) compared to the gelatine gels with added saliva. This phenomenon can be related to the fact that gelatine gels generally have a very smooth surface (Grover et al., 2012), while the surface of sausages might be less smooth. By changing the sausage to a bolus with bolus particle sizes of 8 mm (Fig. 3b), the friction force (F_F) between probe and the bolus was reduced significantly ($p < 0.05$). By increasing the saliva content, the friction force (F_F) further decreased. When the bolus particle size was reduced even more (4.5 mm, Fig. 3a), we can see a significant ($p < 0.05$) influence of saliva concentration on the friction behaviour. In contrast to boli with large particles, saliva addition in small quantities (up to 20% saliva) increased friction forces (F_F). Larger quantities of saliva were required to reduce the friction force (F_F) between probe and the bolus than for the intact sausage. We suggest that especially with the small-sized sausage bolus particles, adhesion between the particles and the probe is of relevance, where saliva is used to hold the particles together in a bolus. The higher friction force for the small particles might relate to the larger total surface area of the small particles, compared to that of larger particles, thus, more adhesion with the upper probe could take place. The adhesion also increases between the food boli particles within the bolus as saliva increases the cohesion and stickiness of the bolus, thereby making it more difficult for the probe to move. These two factors lead to an increase in the friction force. When increasing saliva content even further, friction decreases, as cohesiveness decreases. Another contributing factor might be slip phenomena (Davies & Stokes, 2008). This finding confirms that saliva aids the lubrication of the boli by decreasing adhesion, at sufficient amounts of saliva, thus facilitating swallowing. A comparable observation of such a

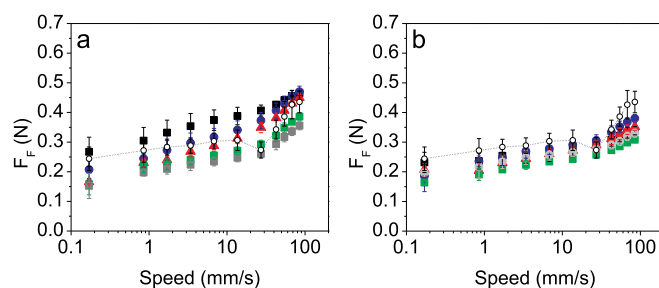


Fig. 3. Friction force (F_F) between a rough PDMS probe and a sausage bolus of a: 4.5 mm particles ($F_{\text{sample}}:23.82$, $p < 0.001$; $F_{\text{speed}}: 66.72$, $p < 0.001$), and b: 8 mm particles, against a PDMS probe with saliva as lubricant ($F_{\text{sample}}:17.8$, $p < 0.001$; $F_{\text{speed}}: 66.36$, $p < 0.001$). Saliva content varied between 5% (black square), 10% (blue circle), 20% (red triangle), 30% (green square) and 40% (grey square). White circles with the dotted line indicate intact sausage (2.5 mL saliva). The dotted line has been added to guide the eye. Average values were obtained from 3 measurements with error bars indicating a 95% confidence interval. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

critical moisture content on the effect of cohesiveness of particulated solids was also shown by research using a ring shear tester (Tobin et al., 2017).

In terms of bolus particle size, we suggest that with an increased surface area, more saliva is used to stick the particles together within the bolus, and less saliva is available to provide lubrication between the bolus and the probe. Thus, more saliva is required to facilitate lubrication. Overall, we can see that the friction behaviour of sausages is (particle) size and lubricant concentration dependent.

These results show that differences in food composition, lubricant content and boli particle size affect the measured friction behavior of intact soft solid foods and food boli, and that the presented set-up is capable of measuring these differences in friction behaviour. Compared to other tribological setups, the strength of this setup lies in its versatility. A large number of intact soft solid foods can be measured, and this method allows to measure the friction behaviour between a probe and soft solid foods directly. In many other tribological setups, the food often has to be present as small particles between two measuring surfaces. Similar to all other setups, a limitation of this methodology is that the friction behaviour is system dependent, which means that the frictional behaviour obtained depends on parameters such as the surface properties of the probe. In a next step, it would be of interest to test and relate the findings made with this setup to results obtained from a sensory study, in order to provide insights in how the friction behaviour of soft solid foods relate to sensory perception.

4. Conclusions

In this short communication, we present a tribological setup to quantify tribological behaviour of intact soft solid foods and food boli. The setup consists of a PDMS probe, applying a defined normal force directly onto soft solid foods. The food is moved in a reciprocal linear motion against the probe and the resulting friction force (F_F) is quantified as a function of speed. Differences in the composition of the food were shown to change the friction behaviour of soft solid foods considerably; present oil droplets in gelatine gels were seen to decrease friction. For food boli, the addition of saliva, as expected, significantly reduced friction, but the amount of saliva required was dependent on the particle size of the boli fragments. We believe that further research in tribology, with continuously improving measurement setups, is valuable to gain more insight into oral processing.

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Declaration of competing interest

The authors have declared that no competing interests exist.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <https://doi.org/10.1016/j.foodhyd.2019.105441>.

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