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Tribology and its growing use towards the study of food oral processing and sensory perception

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1 INTRODUCTION

Tribology is the study of lubrication, friction and wear between surfaces in relative motion. These occur during motion of oral surfaces (tongue, palate, teeth) and arise in the oral processing of food and beverages. Since oral tribology predominantly involves at least one compliant substrate, studies on 'soft-tribology' have emerged to provide knowledge and tools to predict oral behaviour and assess the performance of foods and beverages. Many studies also attempt to relate tribology measurements directly to a food's organoleptic properties, with mixed success. However, a key challenge arises in providing correlations between a complex set of physical measurements with another set of complex sensory measurements, and it is therefore essential to perform studies with exceptional experimental design and confidence in the physical measurement technique. Knowledge in soft tribology provides insight into factors affecting oral sensory perception, including texture, taste, mouthfeel and flavour. Despite the substantial growth in the utilisation of soft-tribological techniques in food science and engineering, there are still challenges associated with interpreting and applying tribological measurements and principles.

We consider it timely to revisit and provide a synopsis of the knowledge base of soft tribology, with consideration for its application towards oral tribology and food lubrication. The following chapter builds upon a previously published chapters and review articles by Stokes and co-authors that provide:

- a detailed introduction to the science of tribology and measurements on food-related systems (Stokes, 2012);
- commentary on how tribology insights influence product design for texture, taste, and flavour (Selway and Stokes, 2014); and

- commentary and review on tribology for studies on oral processing and saliva-food interactions (Pradal and Stokes, 2016).

Here, we provide an overview of the fundamentals of soft contact tribology, measurement systems, surfaces and lubricants including recent developments. Particular emphasis is placed on how tribology is a "system" rather than a material property of a particular lubricant since this is often not realised for those new to the field. In addition, we consider the recent literature concerning food/beverage lubrication and attempts to provide insight into sensory perception of foods and beverages.

2 PRINCIPLES OF TRIBOLOGY

2.1 Tribology is a system property

A key challenge when looking to exploit tribology in analysing food systems is that lubricants (foods, beverages) do not have a signature 'tribology' or 'friction'. This is in direct contrast to the field of rheology; a fluid's rheology is defined in continuum mechanics via constitutive relationships between stress and strain or strain rate. **Tribology is a systems property** rather than a physical property of the fluid (Pradal and Stokes, 2016), and as such it is important to differentiate tribological analysis on the basis of:

- **Measurement system**, e.g. ball-on-disc/pin-on-disc, sliding/rolling/mixture of the two;
- **Surfaces**, e.g. soft/hard/viscoelasticity, hydrophilic/hydrophobic and rough/smooth; and
- **Lubricant** (food), e.g. rheology and heterogeneity, which includes the presence of particles, droplets, air, and surface active ingredients.

To develop suitable tribological measurement techniques that are relevant to oral processing and interpret measurements, it is necessary to take a systematic approach and unravel the complexity of each facet.

2.2 Lubrication regimes for 'Simple' Newtonian Fluids

We class *simple fluids as those that behave as a continuum* within the lubricated contact, whilst Newtonian fluids are those whose viscosity is invariant with applied shear rate. *Hydrodynamic* lubrication arises during entrainment of fluid between nonparallel surfaces due to the generation of a sufficient lift force to support an applied load. When this is also sufficient to deform the substrates, it is referred to as *elastohydrodynamic* lubrication. Both of these lubrication regimes are considered to be representative of *full-film lubrication (FFL)*, and the friction coefficient is the ratio of the shear 'friction' force and normal load. In the FFL regimes, friction is dependent on the substrate deformation and fluid dynamics in the system that are principally governed by the viscosity for Newtonian fluids and elastic modulus of the substrate. Thus it is predicted and generally observed experimentally that the friction force and separation distance between surfaces increases with lubricant entrainment speed under a constant load.

Decreasing the speed of one or both surfaces decreases the lubricant entrainment speed into the contact zone, and as a consequence of decreasing hydrodynamic lift and film thickness between substrates, the friction decreases. When the film thickness decreases to the extent that it is of similar height to surface roughness, the applied load is supported by both the viscosity and substrates, and the friction force increases with decreasing entrainment speed; this is referred to as the *mixed lubrication regime*. The friction force peaks and/or plateaus with further decreases in entrainment speed when the load becomes supported by surface asperities and/or the presence of a boundary layer at the surfaces; this is referred to as the boundary regime.

We generally present the measured friction as a function of entrainment speed in the form of a Stribeck curve, as shown in Figure 1. It is common to present the entrainment speed in the form of a dimensionless number; for example, the Sommerfield number ($\eta UR/W$) or Elastohydrodynamic number ($\eta UE^{1/3}R^{5/3}/W^{4/3}$) (Selway et al., 2017). However, for studies using a constant load (W), radius (R) and substrate (E), it is simply presented as the product of viscosity (η) and entrainment speed (U). This form of the curve is shown in Figure 1 with the three distinct regimes indicated. It should be also noted that the Stribeck curve for simple fluids also depends on the **contact angle** and **roughness** of the substrate (Bongaerts et al., 2007a). While the friction in the full film regime has minimal dependence on the substrate properties, this is not the case for the transition points between regimes nor the friction value in the mixed and boundary regimes.

Figure 1 somewhere near here

We note the full film lubrication regime is well predicted theoretically and computationally for simple Newtonian fluids; this sets up the basis of the *Master curve approach* detailed in the next section. However, in the mixed lubrication regimes, the fluid dynamics, wetting, and substrate material properties (including viscoelastic effects) influence the measured friction. The influence of each of these factors is not easily predicted even for simple Newtonian fluids, as described recently (Selway et al., 2017).

2.3 Lubrication regimes for Complex Fluids: Master curve approach

A key challenge in measuring the tribological performance of food and beverage systems is reconciling measurements with their underlying structure and rheology. To assist interpreting complex materials, we have previously recommended a so-called '**Master Curve Approach**', whereby a Stribeck curve is generated for *simple fluids* with similar wetting characteristics but using fluid with different viscosities to obtain a relationship between friction and the product of speed and viscosity. A master curve is generated by fitting an empirical model to the friction curve (μ_{tot}) using power laws for the boundary (μ_b) and EHL regimes (μ_{EHL}), as follows (Bongaerts et al., 2007a):

$$\mu_b = b(U\eta)^j \tag{1}$$

$$\mu_{EHL} = k(U\eta)^n \tag{2}$$

$$\mu_{tot} = \mu_{EHL} + \left(\frac{\mu_b + \mu_{EHL}}{1+(U\eta/B)^m}\right) \tag{3}$$

b and k are power law coefficients; j and n are power law exponent; B is the value of $U\eta$ below which we have boundary friction, and m is an exponent for the mixed regime.

Creating the master Stribeck curve specific to the tribopair, lubricant and measuring system ensures measurements are accurate and repeatable. More importantly, comparison of master curves generated from Newtonian hydrophilic and hydrophobic lubricants with data from complex foods enables elucidation of the dominant phase in each friction regime. It is expected that data in the hydrodynamic regime should collapse onto the master curve for all fluids as a function of their viscosity (Bongaerts et al., 2007a). In many cases, this is a useful assumption to compare fluid lubrication while excluding viscosity effects.

Soft tribological studies on complex fluids have shown that when they are confined to narrow gaps, the fluid does not necessarily behave as a continuum, and that one or more components (including droplet phases (oil, fat), particulates and hydrocolloids) can have a profound effect on the measured friction response. This occurs in any of the regimes when the film thickness is at the length scale of a component within the fluid, and corresponds to a departure from the master curve for simple fluids. Understanding this effect is critical when examining multicomponent and often multiphase foods and beverages. Examples for such effects on the Stribeck curve for emulsions, particles and polymers are shown schematically in figure 2. For emulsions, in figure 2(a), the schematic shows how the oil-in-water emulsions transition from being dominated by aqueous phase at high speeds and gaps to the situation where the oil phase coalesces in the contact at low speeds. This is dependent on factors such as viscosity ratio, drop size, surfactant and stability (de Vicente et al., 2006a, Stokes, 2014), and is observed in real systems such as creams, mayonnaise, custard and chocolate. For particle suspensions, figure (b), the schematic shows that when the gap is of similar order to a characteristic particle size, particle confinement occurs. This effect has been observed for glass spheres, starch, microgels, insoluble fibres, protein aggregates, and plant cells. (Yakubov et al., 2015a, Chojnicka-Paszun and de Jongh, 2014, Zhang et al., 2017). For hydrocolloids, figure 2 (c), the schematic shows that a decrease in friction arises from both viscosity and adsorption of a polymer-rich film on the surfaces. The viscosity effect is captured using an η_c, the effective viscosity in the contact at shear rates of order 10^3-10^5 s^{-1} where it is noted that the hydrodynamic portion of the polymer curve matches that of the Master curve (Stokes et al., 2011, de Vicente et al., 2005b).

Figure 2 somewhere near here

3 SOFT-TRIBOLOGY MEASUREMENT SYSTEMS

3.1 Instrumentation

Several types of instruments have been used to measure friction in an oral processing context, in a variety of configurations as shown in figure 3, and the instruments and most of the references for each are included below:

- Rolling-ball on rotating-disk ('ball-on-disk') Mini Traction Machine (MTM) PCS Instruments with soft PDMS contact(s), with well-over fifty food oral processing relevant publications (*ca.* 30 publications are included below).
- Rotating ball on three-fixed plates/pins using the Rheo-tribocell attachment for the MCR rheometers (Anton Paar, Gratz, Austria) (Steinbach et al., 2014, Kim et al., 2015, Carvalho-da-Silva et al., 2013, Biegler et al., 2016, Baier et al., 2009, Krzeminski et al., 2012, Sonne et al., 2014a)
- Ring on plate tribo-rheometry using the Discovery Hybrid Rheometer (TA Instruments, USA) on a rough plastic surface of 3M Transpore Surgical Tape 1527-2 (3M Health Care, USA). (Nguyen et al., 2016)
- Optical Tribometer Configuration (Dresselhuis et al., 2007, Dresselhuis et al., 2008a, Liu et al., 2016c, Liu et al., 2016b, Liu et al., 2016a)
- Double-ball-on-plate apparatus attached to MCR rheometers (Joyner et al., 2014b, Joyner et al., 2014a, Joyner et al., 2014c, Goh et al., 2010)
- Plate on three balls (based on a modified texture analyser (TA-XT2, TA Instruments, USA) (Chen et al., 2014, Morell et al., 2017)
- High Frequency Reciprocating Rig (HFRR) PCS Instrument UK (Tsui et al., 2016)
- Rotating shaft with sliding bar (de Hoog et al., 2006, Prinz et al., 2007)

Many of these utilise a rheometer to apply rotation and load to a tribology-fixture attachment. They have emerged in recent years because it is relatively cheap to attach a tribo-fixture to an existing rheometer. They are operated in sliding-only friction, with good normal force control and potential to access a range of speeds (10^{-5}–2300 mm/s, by controlling rotation via torque control) much larger than for the most widely used instrument, the MTM (1–5000 mm/s). However, outputs from these rheo-tribo devices are still challenging to interpret and comprehend, and surface-wear is a common problem although often not reported or considered.

Figure 3 somewhere near here

The technique with the most knowledge base and publications (spanning both tribological and food science/engineering literature) is the commercially available Mini-Traction Machine (MTM) Tribometer (PCS Instruments). The usual configuration is a rotating disk and a ball driven independently to create a mixed rolling/sliding contact. The friction force (F_f) is measured as a function of the entrainment speed $U = (U_{ball} + U_{disc})/2$. The wide range of entrainment speeds enables capture of multiple lubrication regimes and the properties of the surfaces can easily be modified to better mimic in-mouth conditions (Bongaerts et al., 2007a). The slide to roll ratio $SRR = |U_{ball} - U_{disc}|/U$ can be controlled, which is relevant for compliant substrates such as the oral surfaces (Vicente et al., 2006). The key advantage to the use of the MTM over other soft-tribological techniques is that it produces measurements that are normally interpretable due to the depth of the knowledge base that has now been established.

While there are still plenty of knowledge gaps, the MTM has been successfully used to provide quantitative insight into the tribological performance as a function of lubricant rheology (de Vicente et al., 2005a, de Vicente et al., 2006a); surface interactions (Bongaerts et al., 2007a, Myant et al., 2010c, Chojnicka-Paszun and de Jongh, 2014, Selway et al., 2017); emulsions (Douaire et al., 2014), (de Vicente et al., 2006a, Chojnicka et al., 2009); particle suspensions (Yakubov et al., 2015a, Chojnicka-Paszun and de Jongh, 2014, Zhang et al., 2017, Farres et al., 2013, Fernández Farrés and Norton, 2015, Gabriele et al., 2010); hydrocolloids (de Vicente et al., 2006b, Garrec and Norton, 2012, de Vicente et al., 2005b, Stokes et al., 2011, Malone et al., 2003); saliva (Bongaerts et al., 2007b); foods and beverages (Chojnicka et al., 2008, Rodrigues et al., 2017, Chojnicka-Paszun et al., 2012, Selway and Stokes, 2013, Rossetti et al., 2009, Steinbach et al., 2014).

Friction measurements can be combined with complementary techniques such as optical interferometry (Myant et al., 2010a) and fluorescence microscopy (Myant et al., 2010b) to perform film thickness measurements, or Raman spectroscopy to assess the chemical composition of the lubricant present in the contact (Bongaerts et al., 2008). However, these techniques remain underutilised for food systems. A technique that has been used to study oral processes is the optical tribometer cell (OTC), where a detachable upper surface can slide on a lower glass surface mounted on a confocal laser microscope. This device has been used, for example, to elucidate the phase inversion of fat droplets during tribological measurements of emulsions on a pig tongue (Dresselhuis et al., 2008a) and emulsion filled gelatine gels (Liu et al., 2015).

3.2 Surfaces

Surface properties are determinant in the mixed and boundary regimes; therefore, surfaces should be carefully chosen when studying food tribology. Although tribology was traditionally based on steel/steel contacts, the emergence of bio-tribology has pushed researchers to find tribopairs that better mimic *in vivo* situations. From these endeavours, the concept of soft-tribology, where the tribopairs are made of a compliant material has emerged (Bongaerts et al., 2007a, Selway and Stokes, 2014). With non-compliant surfaces and for the typical loads applied between a ball and disc on an MTM instrument (1-5 N load), the resulting pressure is in the order of magnitude of GPa, while by using elastomeric surfaces, the pressure can lowered to 50-100 kPa, which is closer to the 30 kPa found in the mouth (Chojnicka et al., 2008). The current material of choice is polydimethylsiloxane (PDMS), thanks to its ease of fabrication into various shapes and roughness, its tuneable mechanical properties and the possibility to reversibly alter its hydrophobic character (Bongaerts et al., 2007a, Selway and Stokes, 2013, Chojnicka-Paszun and de Jongh, 2014, Chojnicka-Paszun et al., 2014). Rubbers (Chojnicka et al., 2008, Krzeminski et al., 2012) as well as whey protein hydrogels (Joyner et al., 2014a, Joyner et al., 2014c) have been also been investigated, thus providing additional options to match the roughness/viscoelastic properties of the tongue.

To further mimic in-mouth conditions some groups have used a pig tongue on the upper surface of an OTC rather than rubber or silicone (Dresselhuis et al., 2008a, Dresselhuis et al., 2008b). They found that an increase in load resulted in a decrease in friction which they hypothesised was due to the flattening of the papillae on the porcine tongue surface. Above a certain load the papillae are completely flattened, the surface area remains constant and the friction force reaches a plateau. Although this series of studies provides important insight into the behaviour of the tongue in a tribological contact, sample availability and variability might make it impractical to use pig tongue as a wide-spread laboratory tool. As the knowledge of oral surfaces expands, including mechanical properties (Chen et al., 2016, Dresselhuis et al., 2008a, Zhang et al., 2014, Cheng et al., 2011, Chen et al., 2015), composition and mechanism of lubrication (Yakubov et al., 2014, Yakubov et al., 2015b), it is expected that better oral mimetics will become available and allow for more accurate characterisation of the tribology of food products. Only one notable study, by (Ranc et al., 2006), appears to systematically assesses the effect of fabricated surface structures on friction behaviour in a model tribosystem representing the tongue/palate contact. In their investigation, soft silicone samples were fabricated to have well-defined, hexagonal arrays of convex, hemispherical pillar structures to reflect a range of tongue roughness seen in human papillae (Ranc et al., 2006). Friction tests were conducted using a reciprocating motion sliding tribometer, with a hard steel ball used to simulate the palate, under dry conditions, oil, and aqueous solutions (Ranc et al., 2006). Measurements were limited to low sliding velocities (~10mm/s).

The importance of finding surfaces relevant to oral processing is illustrated by the fact that surface roughness (Bongaerts et al., 2007a, Krzeminski et al., 2012, Dresselhuis et al., 2007, Stokes et al., 2011), hydrophobicity (Bongaerts et al., 2007a, Dresselhuis et al., 2007) and modulus (Chojnicka-Paszun and de Jongh, 2014) not only alter the magnitude of the friction coefficient but also the transitions between the different lubrication regimes and the mechanism of lubrication. For instance, roughness changes the mechanism of lubrication in the boundary regime for polysaccharides between soft surfaces, with lubrication on smooth surfaces being a function of hydrated film thickness whereas on rough surfaces it is a function of dry mass of adsorbed polysaccharide (Stokes et al., 2011). In addition, the friction in the boundary-mixed regimes has been shown to decrease with increasing viscosity for very rough surfaces even when using the master curve approach (Selway et al., 2017), which is an important finding when considering the roughness of oral surfaces such as the tongue (see Figure 4).

Figure 4 somewhere near here

4 FOOD AND 'ORAL' TRIBOLOGY

4.1 Lubrication of semi-fluid and fluid foods

Tribological performance of foods is complicated by the multicomponent nature of the food itself, as well as interactions with saliva and oral mimetic surfaces. Food components likely to dominate both mouth feel perception and tribology include particles, fat, emulsifiers and hydrocolloids. However, it is not as simple as the presence or absence of these ingredients but also the influence of in-mouth food structure that can affect tribology (Joyner et al., 2014b). The microstructure of the food product is critical to developing its final form, stability, and mouth-feel (Stokes and Frith, 2008). For example, Liu, Stieger et al. (2015) found that gelled emulsions with unbound oil droplets as the dispersed phase exhibit stronger fat related sensory perception with increased fat content than those with bound oil droplets. This corresponds to lower friction at low speed (boundary regime) for the unbound case. In contrast to ungelled emulsion systems, this finding was independent of the solid fat content. The authors suggest this may be due to the difference in breakdown behaviour of the emulsions and gelled emulsions affecting release of fat droplets to coat the oral surfaces. It is hypothesised that fat lubrication is dominated by oil-surface contact area; this is referred to as the droplet spreading mechanism. Liu, Stieger et al (2015) show for gelled emulsions with low or high fat content the release of oil to the surface governs the measured friction.

Emulsions, containing water and oil with or without an emulsifier have been comprehensively investigated using a wide range of tribometers and surfaces (Joyner et al., 2014b, Dresselhuis, 2008, Dresselhuis et al., 2007, Dresselhuis et al., 2008a, Dresselhuis et al., 2008b, Malone et al., 2003, de

Vicente et al., 2006a). A key driver for tribological behaviour and mouth feel is hypothesised to be coalescence. Coalescence of fat droplets during oral processing leads to an increase in perception of fat-related sensory attributes such as *fatty* and *creamy* and a decrease of measured friction (Chojnicka-Paszun et al., 2012, Dresselhuis et al., 2008a, Dresselhuis et al., 2008b, Dresselhuis et al., 2007). Emulsions can be tuned to the desired stability by altering water or oil phase viscosity, droplet size, emulsifier type and concentration, solid fat content (SFC) and ionic conditions (Joyner et al., 2014b, Dresselhuis et al., 2008b, Liu et al., 2015) . In emulsion lubrication of engineering systems, a desirable outcome is for oil to fill the contact zone in place of the aqueous phase in what is referred to as 'plating out' theory (see review in (Stokes, 2014)).

For simple emulsion systems without emulsifiers, measured using sliding-rolling friction of PDMS-PDMS contacts in an MTM tribometer, the tribological behaviour of the emulsifier-free emulsion system is dependent on the viscosity ratio of the oil and water. When the viscosity of dispersion medium is at least *ca.* 6 times smaller than the dispersed oil, the friction and lubrication film formation is determined by the oil phase. However, when the viscosity ratio is low, the overall behaviour is controlled by the aqueous phase (de Vicente et al., 2006a). Even the relatively simple emulsion systems used to investigate the influence of fat type and structure usually include emulsifies which improve droplet stability and alter surface-fluid interactions. Although emulsifiers are only a small weight percentage of the overall food or model system they can dominate the tribological response due to their ability to adsorb to surfaces (Joyner et al., 2014b). Commonly used nonionic polyoxyethylene surfactants (Tween) reduce the friction between PDMS surfaces, compared to that of water, even when oil is absent (Graca et al., 2007).

Tribological measurement of a fluid cannot be viewed independent of surface properties. Dresselhuis, Klok et. al (2007) show, for sunflower oil in water emulsions stabilised by whey protein isolate, a decrease in friction with increasingly hydrophobic surfaces and rough surfaces. However, Joyner et al. (2014b) show that it is not surface hydrophobicity alone that dictates tribology. They measure protein stabilised emulsions with a double ball on a whey protein isolate ring and show no direct correlation between surface energy or surface contact angle and friction. They conclude that particle size, lubricant viscosity and droplet stability, as well as surface chemistry and mechanical properties all have an influence on tribology. De Hoog et al. (2006) carried out a comprehensive study comparing a range of surfaces with three emulsions: biological surfaces, pig tongue and pig esophagus (inside out); deformable surfaces, rubber; and hard surfaces, glass and metal. They found that biological surfaces display highly complex behaviour where friction is dependent on load and speed due to softness, deformation and roughness of the surfaces. The rubber surfaces were shown to have a smaller dependence on load and speed.

The influence of hydrocolloids is highly dependent on their structure, in particular their melting point in-mouth and their surface interactions. Early work in this area showed that guar gum decreases friction in the mixed lubrication regime as concentration increases, in-line with sensory detection of *slipperiness* (Malone et al., 2003). However, it is critical to factor out the influence on the friction/lubrication due polymer adsorption and from viscosity effects (de Vicente et al., 2006b). Film forming (adsorbing) hydrocolloids - xanthan, gellan and pectin - show two orders of magnitude difference in friction in the boundary regime and decrease in the speed required to enter the full film lubrication regime (Stokes et al., 2011).

The detection of particles in the mouth can be caused by particles in the microstructure of the original food or by particulate structures formed during oral processing. For example, acid denaturation of protein components in saliva results in the sensation of *astringency* or *roughness* (de Wijk and Prinz, 2005). The structure of particles and the food as a whole can influence the detectability in mouth and instrumentally. For example, larger protein aggregates have been shown to result in a decrease of the friction coefficient in the boundary regime and mixed regimes (Chojnicka et al., 2008). Particle interactions with fat droplets and the oral surfaces can influence mouth-feel and instrumentally measured lubrication (de Wijk and Prinz, 2005). A proposed mechanism for this is shown in Figure 5 where there is only a thin film of fluid present or particles are trapped or confined. The presence of particles does not necessarily lead to a negative sensory perception. For example, micro-particulated whey proteins are known to enhance fat related mouthfeel via a ball-bearing lubrication mechanism (Liu et al., 2016c). However, this may not be true for all small particles as Yakubov et al. (2015a) show that this mechanism is dependent on particle surface interactions encouraging or preventing particle entrainment. Sonne et al. (2014b) suggest that the dominant phase (particles or matrix) is dependent on the gap between tongue and palate.

Figure 5 somewhere near here

4.2 Saliva and Saliva-food interactions

Saliva serves several essential functions such as protection of the oral mucosa, pH maintenance, microbial control, remineralisation of the teeth, lubrication, swallowing, bolus formation, digestion and taste mediation. (Kaplan and Baum, 1993, Humphrey and Williamson, 2001, Carpenter, 2013, Valdez and Fox, 1993). Normal flow of saliva is critical to chewing and swallowing (Engelen and Van Der Bilt, 2008) and the composition of the saliva also influences taste and texture perception, possibly through the effects of dilution, breakdown by enzymes, or changes in the lubrication due to interactions between salivary and food components (Neyraud et al., 2012, Engelen et al., 2007, Engelen and Van Der Bilt, 2008, Rossetti et al., 2008, Selway and Stokes, 2014). Saliva is a good lubricant that results in a boundary friction coefficient of order 0.01 at smooth PDMS surfaces, which is two orders of magnitude lower than that of water in the same conditions (Bongaerts et al., 2007b, Macakova et al., 2011). Although the mechanism by which saliva forms a lubricant and wear resistant boundary film on oral surfaces is not fully elucidated, it is thought that it is based on a multilayered architecture, comprised of a dense base layer of small molecular weight proteins (proline-rich proteins, histatins, statherins and others) surmounted by larger glycoproteins, called mucins, assembled in loops and trains. This multi-layered structure, by ensuring the formation of a thick (~100 nm) hydrated layer, might be essential to the lubrication process (Macakova et al., 2011, Gibbins et al., 2014, Yakubov et al., 2015b).

Interactions between food components and saliva can lead to changes in the salivary film lubrication. (Figure 6). These could be due to changes in pH or ionic strength, that alter the conformation and hydration of the salivary film (Macakova et al., 2010, Macakova et al., 2011, Selway and Stokes, 2013). Salivary protein aggregation and the subsequent mechanical desorption of the salivary film under the influence of the load is another mechanism for changes in the lubrication of a pre-adsorbed salivary film upon addition of food or beverages (Selway and Stokes, 2013, Rossetti et al., 2009, Vardhanabhuti et al., 2011). These changes in salivary lubrication may lead to changes in perception, although the link is yet to be fully established. Nevertheless, these effects all point to the importance of incorporating saliva, either by pre-mixing saliva with the food sample or by observing the response of a pre-adsorbed salivary film upon addition of food products, into any predictive instrumental measurement and model for texture perception

Figure 6 somewhere near here

5 LINKING TRIBOLOGY MEASUREMENTS TO SENSORY ATTRIBUTES

Sensory perception is a complex area requiring clearly defined parameters and trained panellists to minimise variability and ensure objective measurement. Removing confounding factors such as taste, aroma and visual is also critical to texture perception (Engmann and Burbidge, 2013). However, just as thin film coatings on oral surfaces are anticipated to affect mouthfeel and texture, they also influence mass transfer that is key factor in taste and aroma perception (Selway and Stokes, 2014). Despite the anticipated interconnectedness between taste, aroma and mouthfeel/texture, we focus here on only two sets of sensory attributes that have been the main focus of tribology and sensory measurement; creamy-texture and astringency. However, we note that a foods sensory attributes are also complex and multidimensional because:

- foods are rheologically complex, heterogeneous and contain multiple components;
- oral substrates (teeth, mucosa, tongue) and oral fluids (saliva) are complicated materials and responsive to their environment;

- cross-modalities arise during sensing and transduction to cognitive processing in the brain.

The complexity of the multiscale tribological behaviour on sensory is captured well by figure 5 from de Wijk and Prinz (2005).

5.1 Yoghurts, creams, fat suspensions: *creamy* perception

Creaminess, a sensory descriptor used with a broad range of foods, is closely related to consumer acceptability and liking (Elmore, 1999). C*reaminess* is a complex multimodal food texture that is difficult to quantify due to influences from colour, aroma, flavour, and other textural attributes (Rao, 2014, Chojnicka-Paszun et al., 2012), as well as both bulk and surface properties (de Wijk et al., 2006, van Vliet et al., 2009, Pascua et al., 2013). Because of this it is difficult to correlate this property directly to physical measurements of food products and a range of measurements are used encompassing bulk, surface and frictional properties (Pascua et al., 2013, Chen and Stokes, 2012, Sonne et al., 2014b). With the ever-increasing pressure on food manufacturers to produce low fat products with acceptable mouth-feel, tribology has become a key for gaining an instrumental measure of *creaminess-texture*.

There is a significant volume of work correlating the amount of fat with both friction and sensory perception in dairy products. Chojnicka-Paszun, de Jongh et al (2012) performed a useful study that capture the lubrication and sensory properteis of homogenised milk with fat contents ranging from 0.1 to 8%. They found a linear correlation between percieved *creaminess* mouthfeel and friction at fat content above 1%, using the friction coefficient measured at speeds of 10 mm/s on silicone rubber (o-ring) surfaces. They suggest correlation arises from droplet coalescence on the surfaces under specific conditions, but no data was provided to support this claim and the friction observed is not conmesurate with that expected for the fat phase alone. We suggest that the key driver for the correlation is *likely* the viscosity of the milk samples; the correlation coefficient between the viscosity and creamy mouthfeel was similar to that for fricton, and model studies show that the measured friction in the boundary-mixed regime for rough viscoelastic surfaces (relevant to the surfaces of Chojnicka-Paszun, de Jongh et al (2012)) is a function of viscosity (Selway et al., 2017). Recent work also shows a strong correlation between fat percentage and friction measurements on a Discovery Hybrid Rheometer using ring-on-plate tribo-rheometry (TA Instruments, USA) on a rough plastic surface of 3M Transpore SurgicalTape 1527-2 (3MHealth Care, USA) (Nguyen et al., 2016). The measured friction appears to be in the boundary-mixed lubrication regime, and in the absence of a substantiated mechanism, we speculate this dependence on fat content arises from viscous effects in line with the multiscale viscoelastic lubrication mechanism examined by Selway et al. (2017). Le Calve, Saint-Leger et al. (2015) show that for yoghurt the relationship between friction and creaminess perception is non-linear and in agreement with their sensory results. Sensory discrimination was possible between 0.5% and 1.75%, which corresponded to a difference in friction coefficient of 0.02 at an entrainment speed of

0.01 m/s. However, it was not possible to discriminate between 1.75% and 3.5% fat, which corresponded to a small difference in friction coefficient of 0.005 at 0.01 m/s.

Several results in the literature emphasise the potential of tribology as a predictive tool by providing evidence for a correlation between friction, fat content and sensory texture. However, whilst promising and very insightful, we recommend caution in using tribology as a creaminess correlator at this stage, because the studies typically do not provide a compelling mechanisms for observed tribology measurements or the correlation with sensory. It is challenging to determine these mechanisms on the basis of existing literature as few incorporate systematic design and measurement in the three key areas of: *known product structure*; a suite of *instrumental measures* with supporting fundamental basis; and *simple model systems* which eliminate cross-modal effects and where factors (e.g. viscosity and fat content) are tuned independently.

Complex dairy systems, including custard, have been used to identify confounding factors and the influence of saliva. Saliva dilutes the food system and α-amylase from saliva causes hydrolysation of starch, which results in loss of structure and viscosity. It is hypothesised that loss of both structure and viscosity contribute to a reduction in creamy after-feel due to a thinner, less viscous coating on the tongue (Engelen et al., 2007). In one study, a lower friction was observed for a custard in an amylase solution in comparison to one mixed with the same volume of water. Despite the reduction in viscosity caused by dilution with saliva and breakdown of starch by amylase, which should lead to an increase in friction, the authors hypothesise that during breakdown of the starch matrix fat is released reducing friction and increasing the sensation of *creaminess* (de Wijk and Prinz, 2005). This was particularly important in low fat products where, prior to amylase addition, little fat was available for coating the oral surfaces (de Wijk and Prinz, 2005).

5.2 Tribology and perception of astringent compounds

Astringency is described as the drying-out and puckering sensation that follows consumption of certain food or drinks such as red wine, tea or fruit (Upadhyay et al., 2016). Although astringency can be desirable and even expected in some instances, for example in red wine, high levels of astringency can lead to an unpleasant feeling and ultimately low consumer acceptance of the food product. Polyphenols (Soares et al., 2015), multivalent cations (Lim and Lawless, 2005), positively charged proteins and polysaccharides (Luck et al., 2015, Vardhanabhuti et al., 2010) can elicit an astringent sensation. At the molecular level, the binding and precipitation of salivary proteins with astringent compounds is thought to form the basis for astringency, although the exact molecular mechanism and how it relates to the sensory perception is still unclear and is thought to vary with the nature of the astringent (Gibbins and Carpenter, 2013, Poncet-Legrand et al., 2006, Lee et al., 2012). More specifically, the binding of proline rich proteins (PRPs) with polyphenols, which have recently been highlighted as playing an important role in the anchoring of the salivary film (Yakubov et al., 2015b), is thought to be important in the astringency perception of polyphenolic compounds (Jöbstl et al., 2004, Baxter et al., 1997). The complexation process and the nature of the PRPs interacting with the phenolic compounds can be influenced by the presence of carbohydrates (Soares et al., 2012), concentration of alcohol (McRae et al., 2015) and nature and concentration of the tannins (Soares et al., 2011b, Soares et al., 2011a). PRPs are not the only proteins complexing and precipitating under the influence of astringent compounds: for instance mucins have also been shown to precipitate in the presence of polyphenols (Davies et al., 2014), beta-lactoglobulin (Vardhanabhuti et al., 2010) chitosan (Luck et al., 2015) or cationic compounds (Biegler et al., 2016).

Because astringency is often described as a loss of lubrication, tribology appears as a tool of choice to probe its macroscopic manifestations and provide the missing link between the molecular and sensory levels. The conformational changes highlighted by the molecular study of astringency point to a possible link with the disruption of the salivary film lubrication. Therefore, the tribological response of the salivary film upon addition of astringent compounds has been the main focus of the studies in this area. In a recent study, loss of lubrication was shown for saliva and purified salivary mucins upon exposure to a series of cationic astringent compounds (chitosan, $Fe_2(SO_4)_3$, $AlCl_3$ and lysozyme) (Biegler et al., 2016), which was attributed to mucin complexation with the astringent compounds to various degrees. In this case, tribology appeared as a promising tool to study astringency. However, other studies have revealed that distinctions need to be made: Rossetti et al. used a MTM tribometer with a salivary film pre-adsorbed onto PDMS surfaces to study the effect of three astringent compounds: epigallocatechin gallate (EGCG), epicatechin gallate (ECG) and epicatechin (EC) (Rossetti et al., 2009). Although a clear loss of salivary lubrication occurred upon addition of EGCG and ECG to a salivary film, EC did not provoke such change although it was perceived as astringent. In a different experiment, they found that the decrease in astringency perception obtained by mixing maltodextrin with ECGC was accompanied by a decrease in friction. However, whilst addition of milk to ECGC decreased astringency, it led to an increase in friction that corresponded to an even greater loss of the salivary film than ECGC alone. A similar experiment showed that the pH dependent astringency of β-lactoglobulin could be linked to a loss of lubrication of saliva, however no clear link could be observed between the concentration of the astringent compound and the friction measurements (Vardhanabhuti et al., 2011). These results indicate that although in some cases astringent compounds can lead to a loss of salivary lubrication measured using tribology, a more complex and subtle mechanism might be at play. Further research is needed to establish complementary techniques that could be used in conjunction with tribology to predict the astringency of food compounds *in vitro*. That is, the measurement is useful to study saliva-beverages/food interactions, but a loss of salivary lubrication doesn't necessarily equate to astringency (Rossetti et al., 2009).

6 CONCLUDING REMARKS AND OUTLOOK

There is now an abundant set of literature on the behaviour of a wide variety of lubricants in soft-tribological contacts, including model soft-solids, complex fluids (polysaccharide and protein solutions), and heterogeneous systems (emulsions, suspensions) as well as foods and beverages. This foundation of understanding on their lubrication behaviour has principally been established in soft-tribological studies involving sliding/rolling ball-disk set up on mini-traction machines utilising relatively smooth hydrophobic surfaces; the insights obtained can be rationally applied to food and beverage design.

Tribology-sensory relationships exist for many foods/beverages, but they are restricted to the specific formulations and tribological configuration utilised, and cannot usually be applied more broadly. With a careful and rigorous formulation/experimental design, we envisage tribological tools to provide insights into the sensory perception of foods in combination with other *in vitro* techniques such as rheology, particle sizing or characterisation of surface interactions. The ingredients for the successful use of tribology in the prediction of sensory attributes include: well characterised tribopairs and equipment; a careful characterisation of simpler model foods before considering complex food products; the incorporation of saliva in tribological studies; the removal of confounding factors from the sensory study and a global approach that considers all regimes of lubrication; and the intervention of other physico-chemical factors. To utilise outcomes for rational design, it is essential to ensure that the mechanism for instrumental tribology measurements is consistent with the in-mouth behaviour leading to the sensory response. In addition, creating and validating substrates that mimic the roughness and mucosal-film coating of oral surfaces is also highly desirable and worthwhile activity for future research. Provided these complexities are considered appropriately, we anticipate that the field of tribology in an oral processing context will continue to grow and ultimately that it will provide valuable methods for rational design.

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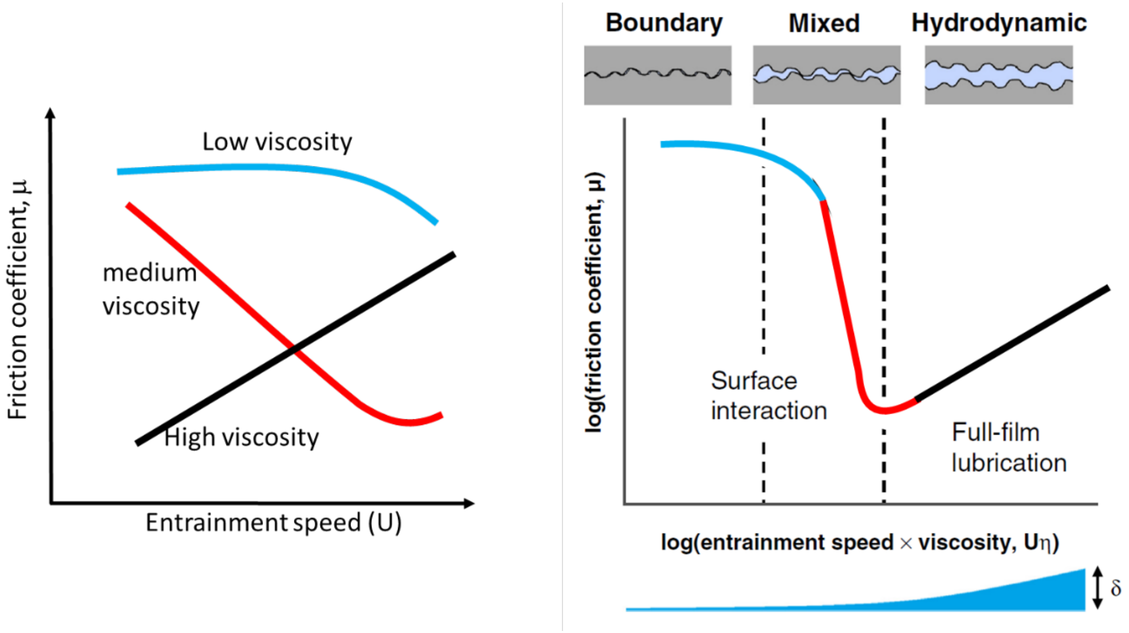

Figure 1 – Schematic of the Stribeck curves for 3 different Newtonian fluids in a soft-tribological contact. The right hand figure (reproduced with permission (Selway and Stokes, 2013)) demonstrates the use of the Master curve approach to combine 3 Newtonian fluids (usually glycerol-water or sugar-water mixtures) onto a single "Master" curve with three lubrication regimes shown.

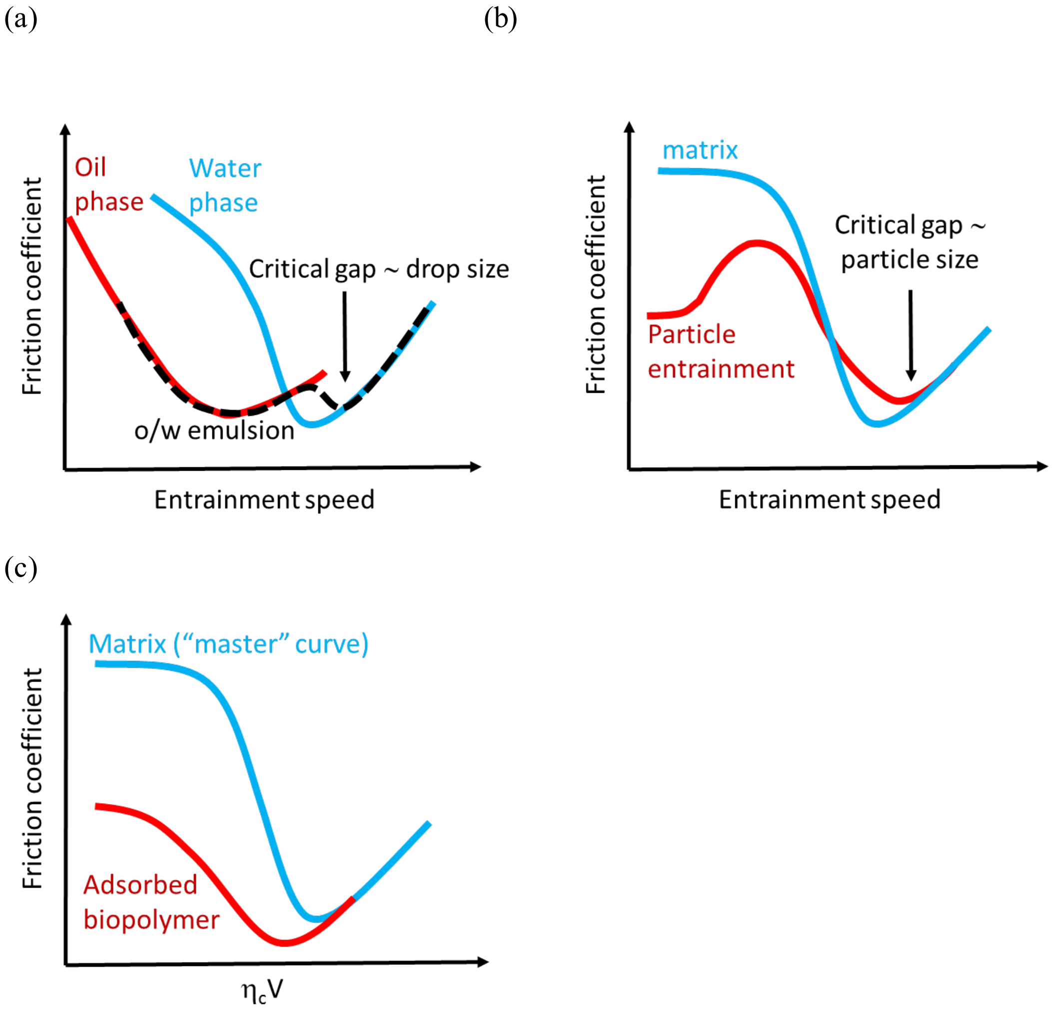

Figure 2 Schematic of typical Stribeck curves arising for (a) emulsions (b) particle suspensions and (c) adsorbing biopolymers (hydrocolloids) lubricated within soft-tribological contacts. For emulsions (a), the schematic shows how the oil-in-water emulsions transition from being dominated by aqueous phase at high speeds and gaps to the situation where the oil phase coalesces in the contact at low speeds. For particle suspensions (b), the schematic shows that when the gap is of similar order to a characteristic particle size, particles confinement occurs. For hydrocolloids (c), the schematic shows that a decrease in friction arises from both viscosity and adsorption of a polymer-rich film on the surfaces.

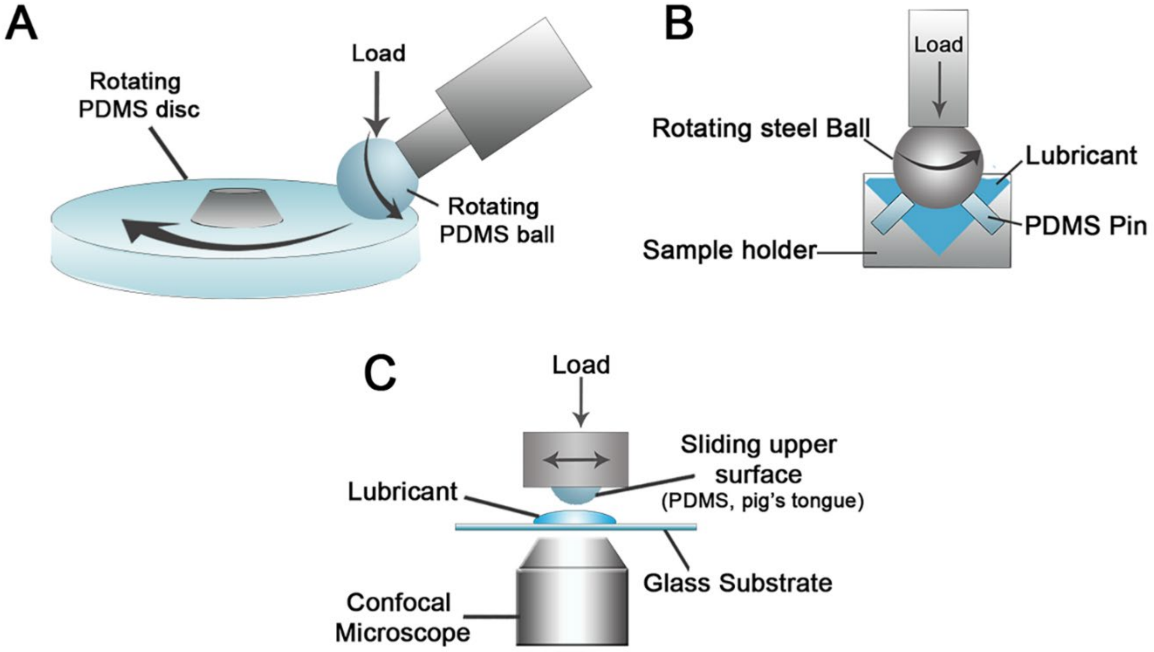

Figure 3 - Schematics of commonly used tribology measurement equipment. PDMS is used as an example but other materials can be used. (A) Mini-Traction Machine in ball on disc configuration (B) Anton-Paar ball on three pins rheometer attachment (C) Optical Tribometer Configuration.

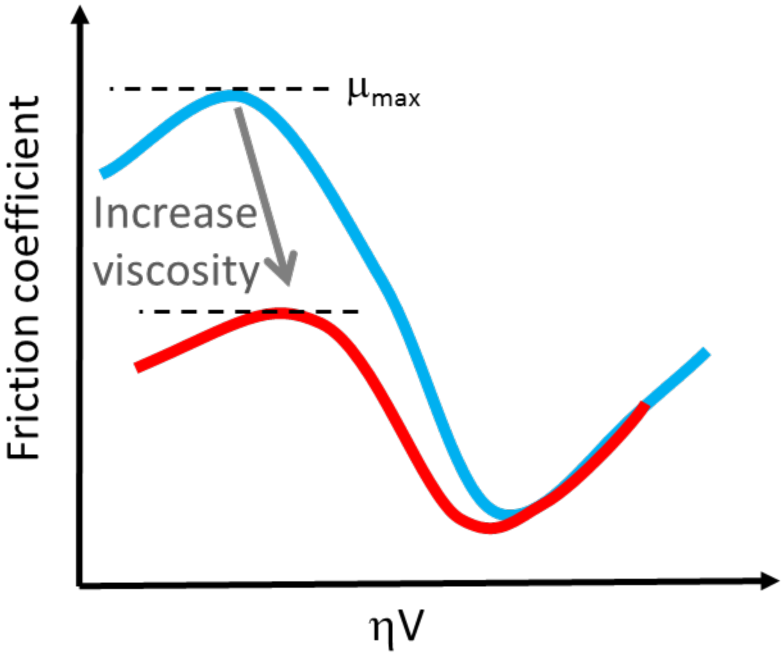

Figure 4 – Schematic showing how the peak viscosity at the boundary-mixed transition region decreases with increasing viscosity, based on (Selway et al., 2017). This effect of viscosity was demonstrated on a range of different tribopairs including rough PDMS substrates.

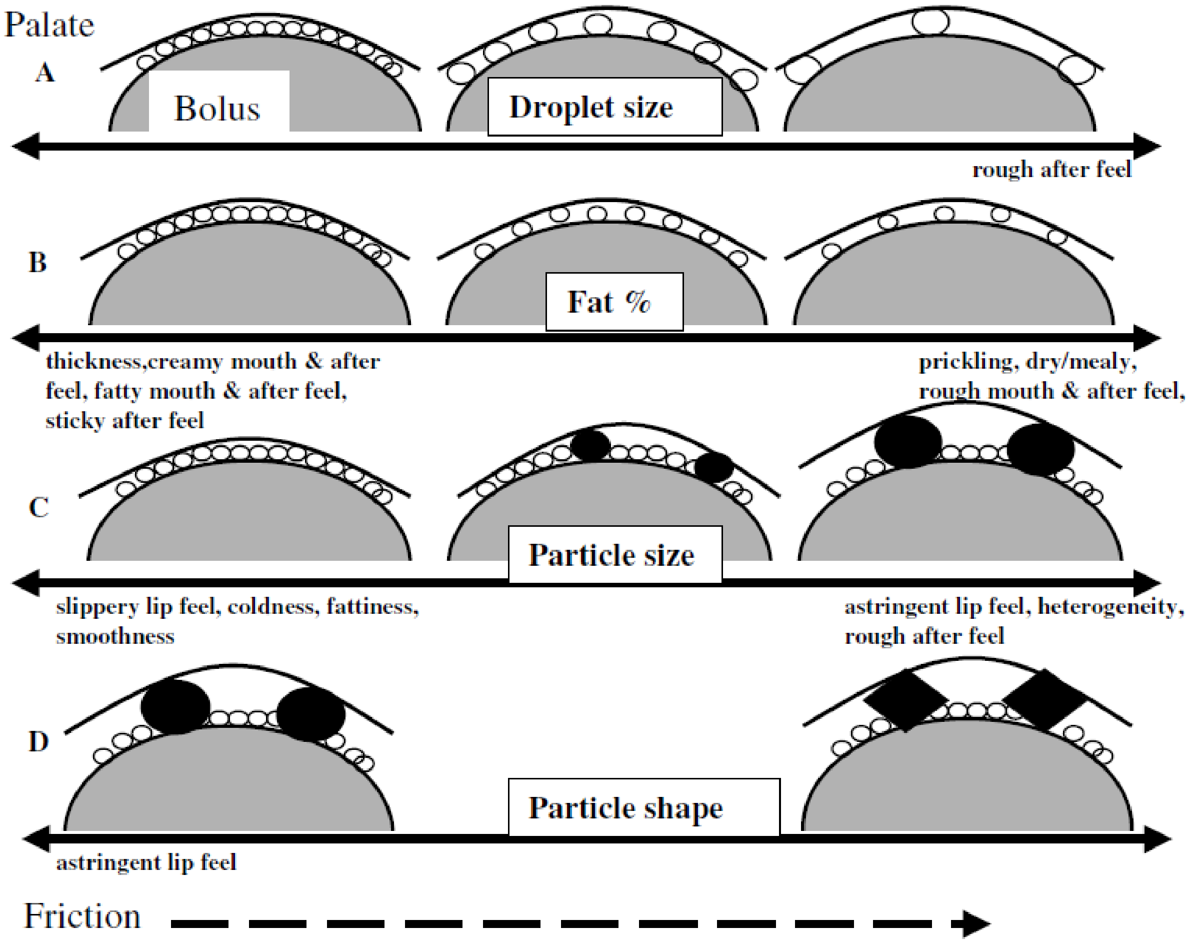

Figure 5 – Schematic diagram reproduced with permission (de Wijk and Prinz, 2005). Showing the effect of droplet size, fat % and particle size and shape on friction. Grey indicates the food, white is fat droplets and black is solid particles.

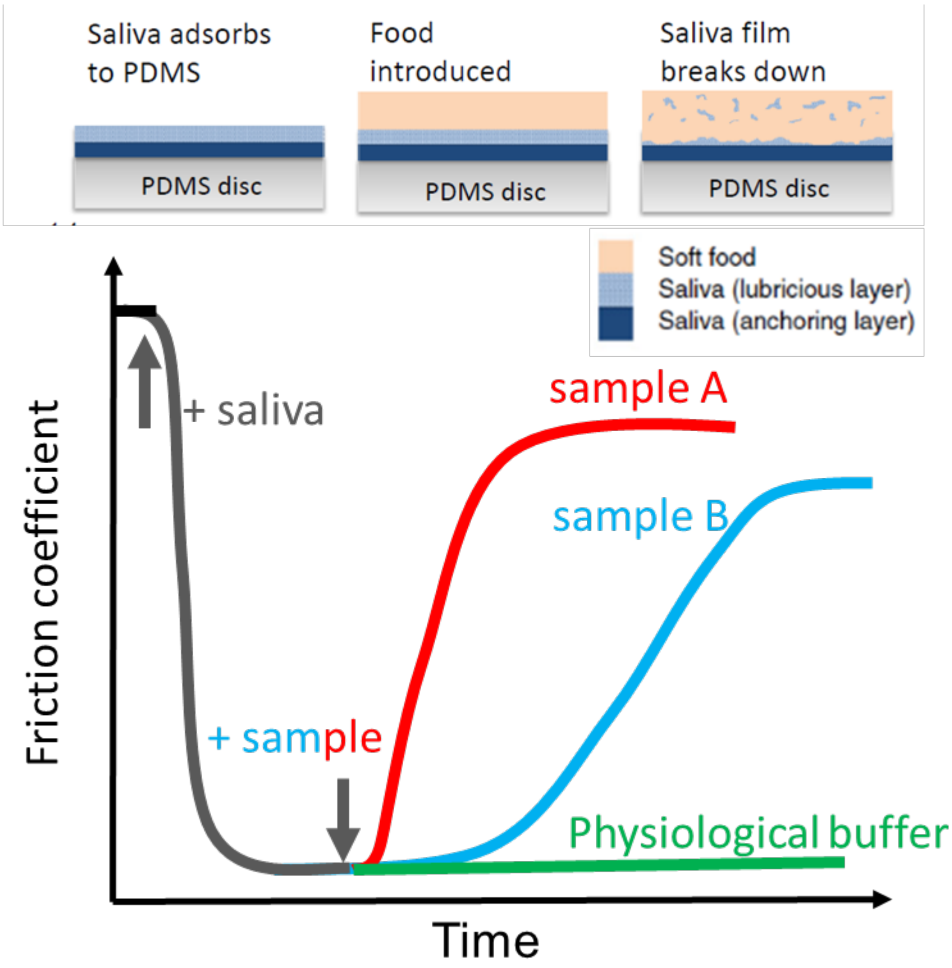

Figure 6 - The schematic shows a friction coefficient being measured at a constant speed and load with the systematic addition of saliva and test sample. Saliva is highly lubricating and absorbs to form a viscoelastic film on the surfaces and decreasing the friction coefficient from an initial value of ca. 1 (black line) to ca. 0.01 (grey line). Upon addition of sample, sample A (red line) causes a rapid increase in friction that occurs if saliva detaches from the surface, while sample B (blue line) affects the film much more slowly. In comparison, addition of a physiological buffer (green line) has no significant effect on the friction, indicating that it arises from a hydrated adsorbed polymer film that is bound to the surface (Macakova et al., 2011, Macakova et al., 2010). These effects are observed in studies on beverages (Rossetti et al., 2009), yogurts, custards (Selway and Stokes, 2013).