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Oral tribology: providing insight into oral processing of food colloids

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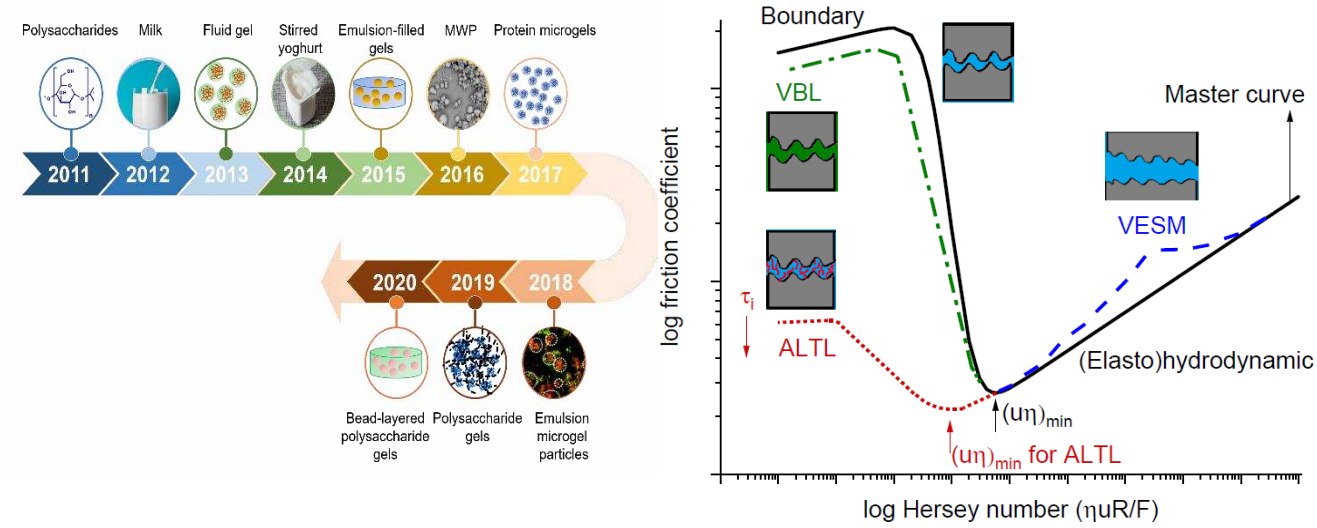
Highlights

- Evolution of use of oral tribology in food colloid science is presented
- Key lubrication theories relevant to complex food fluids are covered
- Current applications of tribology specific to food colloid research are discussed
- Advanced complementary techniques to uncover frictional dissipation are examined
- Tribology may help addressing food safety/ sustainability challenges in the future

Graphical Abstract



Abstract

Food oral processing research has attracted a great deal of attention in the last few decades owing to its paramount importance in governing sensory appreciation and pleasurable experience of consuming foods and beverages and eventually regulating nutrient intake. A range of physiologically-controlled unit operations from first bite, to particle size reduction, mastication with saliva, bolus formation, swallowing to generation of oral residues occur during oral processing of solid foods across a range of time and length scales resulting in various physical, biochemical and psychological consequences. Tribology is the study of friction and lubrication, and it has emerged as a key tool to deconvolute these complex processes and provide insights into the physics of oral processing and sensory perception. This review provides an overview of "oral" tribology including experiments in the last decade on food colloids and lubrication theories, and highlights a perspective on how the field is evolving to address various food science challenges. This includes current and forecast future applications of tribology in designing healthier foods, creating sustainable alternatives without compromising mouthfeel, tackling food counterfeiting and tailoring foods for aging populations. We also examine a suite of advanced complementary techniques such as imaging, scattering, and adsorption using specific examples from allied fields that would be useful to uncover frictional dissipation in food systems in the future. The perspectives provided in this review thus represent an exciting glimpse of what tribology can offer, which may become a routine and integral step in food colloidal design in the future.

Keywords: Oral tribology; Stribeck curve; lubrication; biopolymer; rheology; adsorption

1. Introduction

Historically, food colloid science has been dedicated to developing structure-property relationships controlling the stability of colloidal ingredients and complex multiphasic food structures during processing and storage. Due to the continuous rise in food-linked diseases, such as obesity and cardiovascular diseases, there has been an upsurge in research interests in the colloids community over the last few decades to investigate interaction of food colloids with various physiological sites within the human body. There is now a consensus that food undergoes a series of complex transformation across a range of time and length scales in the mouth upon consumption. In addition, the heat and mass transport phenomena occurring in mouth influence the dynamic hedonic appreciation (Chen, 2009; Selway & Stokes, 2014). The multi-scale transformation of food and the oral transit time not only affect the psychological aspects of sensory perception but also impact the physiological biomarkers governing nutrient intake and oro-sensory satiety (Campbell, Wagoner, & Foegeding, 2017; Hetherington, 1996; Krop, Hetherington, Miquel-Kergoat, & Sarkar, 2018; Stribiţcaia, Evans, Gibbons, Blundell, & Sarkar, 2020a). Particularly, the change in length-scale *i.e.* the film thickness of food/ food-salivary film between the oral surfaces is proposed as a key factor. It is postulated that oral processing involves gradual transition of regimes where the bulk fluid properties govern the sensory outputs such as thickness, to one where surface interaction-induced tribological properties tend to dominate the mouthfeel perception, such as smoothness (Chen & Stokes, 2012; Kokini & Cussler, 1983; Pascua, Koç, & Foegeding, 2013).

Food oral processing of solid foods includes six essential unit operations ranging from first bite, comminution, granulation involving food particle-saliva as well as food particle–surface interactions, bolus formation, swallowing to post swallowing generation of oral residues, and the transformation of food during this sequence is suggested to relate to the temporal sensory perceptions of texture, taste and aroma (Stokes, Boehm, & Baier, 2013).

Although oral processing research has existed for several decades (Hutchings & Lillford, 1988; Prinz & Lucas, 1997), the timely review by Chen (2009) summarizing the oral physiology and rheological aspects of food in the oral regime as well as swallowing helped stimulate a renaissance in research interests in food oral processing among the colloid scientists.

If we dissect the food oral processing research domain, it includes four main subareas, 1) oral behaviour including chewing (Brown, Eves, Ellison, & Braxton, 1998; Foster, et al., 2011; Prinz & Lucas, 1997; Wilson, Luck, Woods, Foegeding, & Morgenstern, 2016) 2) food bolus rheology (Devezeaux de Lavergne, van de Velde, & Stieger, 2017; Foegeding, et al., 2011; Nishinari, Fang, & Rosenthal, 2019; Stokes, et al., 2013; Witt & Stokes, 2015), 3) food-saliva interaction (Mosca & Chen, 2017; Sarkar, Ye, & Singh, 2017b), and 4) sensory perception (Chen, 2014; Palczak, Blumenthal, Rogeaux, & Delarue, 2019; Pascua, Koç, & Foegeding, 2013; Schlich, 2017), of course, not in any particular order. In addition, the allied areas impact of food oral processing on food intake/ satiation/ satiety and, as well as role of oral processing in nutrient digestion (*in vitro* and *in vivo* studies) and their metabolic responses. However, a paradigm shift in the field of oral processing to include the fifth indispensable aspect *i.e.* oral tribology involving the study of oral friction, lubrication and wear can be noticed now with exponential growth of research output numbers and citations.

The pioneering work nearly 44 years ago (Kokini, Kadane, & Cussler, 1977) highlighted the importance of oral tribology to govern some of the key organoleptic properties such as smoothness, slipperiness, which could not be described by instrumental rheological characterization alone. In the early 21st century, this work has been sporadically followed by tribology of saliva (Bongaerts, Fourtouni, & Stokes, 2007), chocolates (Lee, Heuberger, Rousset, & Spencer, 2004), tea (Rossetti, Bongaerts, Wantling, Stokes, & Williamson, 2009), model hydrocolloids particularly in the fluid film regimes (de Vicente,

Stokes, & Spikes, 2006), nonionic polyoxyethylene surfactants (Graca, Bongaerts, Stokes, & Granick, 2007) and emulsions (Malone, Appelqvist, & Norton, 2003). Nevertheless, the last ten years captured in **Figure 1** shows a systematic progress in oral tribology of food colloids and colloidal ingredients from various research groups across the globe focusing on food polysaccharides, proteins, gels, microgels and fluid gels, emulsion microgels to foods such as yoghurts, cheese *etc.* Of course besides these major developments highlighted in **Figure 1**, elegant progress has been made in tribological understanding of complex emulsions (Douaire, Stephenson, & Norton, 2014; Oppermann, Verkaaik, Stieger, & Scholten, 2017), iso-rheological semi-solid colloids (Laguna, Farrell, Bryant, Morina, & Sarkar, 2017a; Selway & Stokes, 2013) and solid foods (Kim, et al., 2020). Therefore, this appears as an opportune time to write a review on oral tribology of food colloids in this 35th Anniversary Issue of *Food Hydrocolloids*.

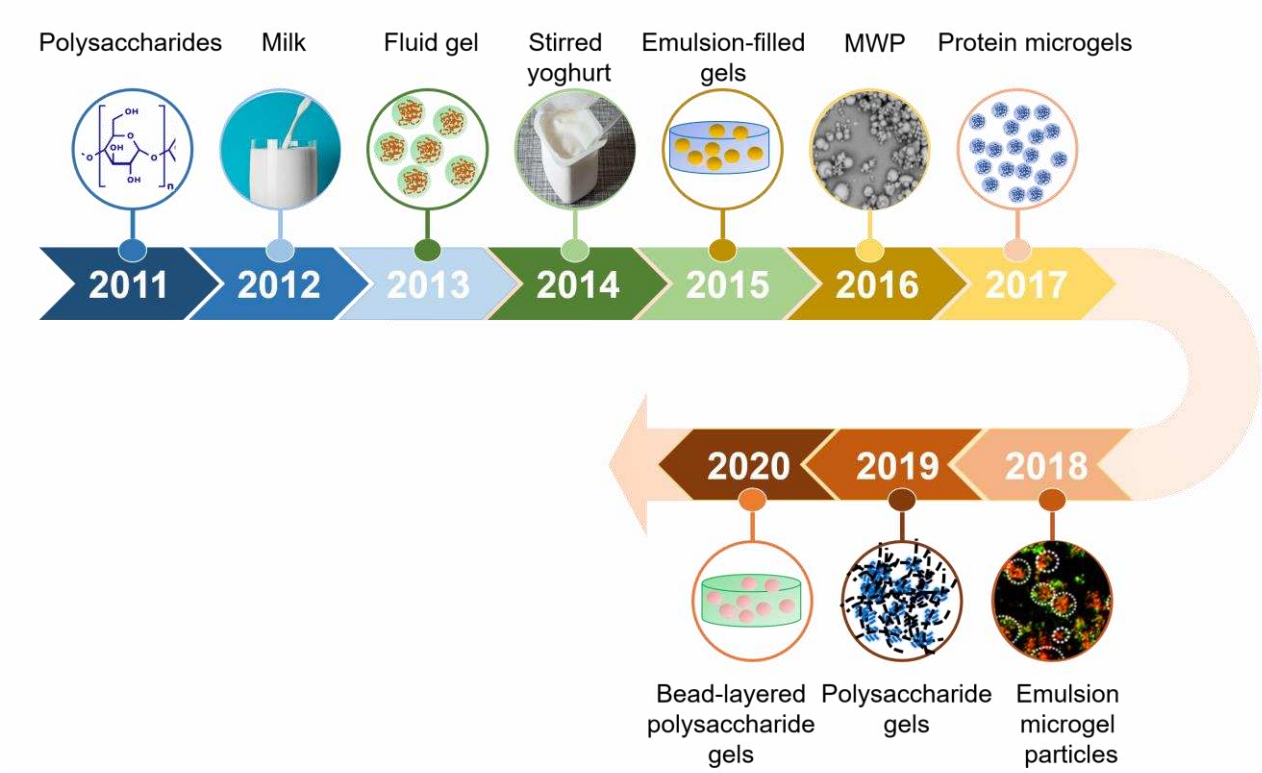


Figure 1. Major developments in tribology of colloidal ingredients, model systems and real food in the last ten years. The development in each year is considered to be major depending upon the new insights generated in tribology of colloid (with > 20 citations in Google Scholar) except the 2020 article. Any development on new tribological device or surface is not included. The timeline shows tribological characterization of

polysaccharides, such as pectin, xanthan gum, gellan, and locus bean gum (Stokes, Macakova, Chojnicka-Paszun, de Kruif, & de Jongh, 2011), milk with a fat content ranging between 0.06 and 8 wt% (Chojnicka-Paszun, de Jongh, & de Kruif, 2012), *κ*-carrageenan-based fluid gel (Garrec & Norton, 2013), stirred yoghurt testing effects of fat, protein, and casein to whey protein ratio (Sonne, Busch-Stockfisch, Weiss, & Hinrichs, 2014), fat droplets dispersed in emulsion-filled gels (Liu, Stieger, van der Linden, & van de Velde, 2015), microparticulated whey protein (MWP) dispersed in liquids or gels (Liu, Tian, Stieger, van der Linden, & van de Velde, 2016), whey protein microgels testing effects of volume fraction and surfaces (hydrophilic/ hydrophobic) (Sarkar, Kanti, Gulotta, Murray, & Zhang, 2017a), emulsion microgel particles (Torres, Andablo-Reyes, Murray, & Sarkar, 2018), polysaccharide gel boli with model saliva (Krop, Hetherington, Holmes, Miquel, & Sarkar, 2019) and bead-layered hydrogel boli with model saliva (Stribiţcaia, Krop, Lewin, Holmes, & Sarkar, 2020b).

The present review thus focuses on the oral tribology of food colloids with a clear emphasis of providing perspectives on future research. Oral tribology has a huge potential for designing biophysically informed food colloids in the future in a cost-effective manner, but effective mimicking of the complex features, deformability and motions of oral surfaces to perform tribological experiments and harmonization of such surfaces to answer specific questions are still needed to enable its widespread use. Although there has been growing interests in designing new tribometers with continual attempts to emulate oral tongue surface in terms of softness, wettability and roughness (Andablo-Reyes, et al., 2020; Bongaerts, et al., 2007; Carpenter, et al., 2019; Rudge, Scholten, & Dijksman, 2020; Sarkar, et al., 2017a; Taylor & Mills, 2020) using a variety of tribological or custom-made rheological set-ups or altering motions in the set ups (Andablo-Reyes, et al., 2020; Fuhrmann, Aguayo-Mendoza, Jansen, Stieger, & Scholten, 2020; Mo, Chen, & Wang, 2019; Tsui, Tandy, Myant, Masen, & Cann, 2016; Wang, Wang, & Chen, 2021), such designs and improvements in tribological set ups are considered to be beyond the scope of this article. We also do not cover salivary tribology or food-saliva interactions in this article as this topic has been extensively covered in recent reviews (Boehm, Yakubov, Stokes, & Baier, 2020; Mosca & Chen, 2017; Sarkar, Andablo-Reyes, Bryant, Dowson, & Neville, 2019a; Sarkar & Singh, 2012; Sarkar, Xu, & Lee, 2019b; Sarkar, et al., 2017b). The comprehensive reviews on rheological to tribological transition during oral processing and analysis of various lubrication regimes as well as growing importance of tribology to study food oral processing (Chen & Stokes, 2012; Sarkar, et al., 2019a; Selway & Stokes, 2014; Shewan, Pradal, & Stokes,

2019) as well as reviews on tribology-sensory relationship (Pradal & Stokes, 2016; Prakash, Tan, & Chen, 2013; Sarkar & Krop, 2019; Stokes, et al., 2013) facilitate this current article.

Here, we aim to provide a different perspective to previous reviews by focusing on the theories of lubrication and soft tribology, and highlighting some important models that are seldom used to discuss experimental tribological results in the food community. We anticipate that this section on lubrication theories should enable making quantitative elucidation of tribological regimes and lubrication mechanisms of complex food fluids, which often get underestimated in food colloid science literature. We also direct our attention to how tribology can be used to address various food-linked challenges ranging from designing healthier colloids, addressing food sustainability, detect food counterfeiting as well as designing food for vulnerable population using specific case studies from literature. Finally, we examine several ancillary techniques drawing inspiration from other fields that could benefit unravelling various lubrication mechanisms occurring in complex multiphasic food colloidal systems. We hope the article written in an opinion style particularly outlining the future of tribology in food applications will entice colloid scientists to use tribology as a mechanical tool answering mechanistic questions behind the sensory appreciation of food.

Table 1. List of abbreviations and symbols.

Acronyms	Full form	Symbols	Definition
AFM	Atomic force microscopy	h_{min}	Minimum lubricant film thickness
ALTL	Adsorbed lubricious thin layers	E'	Equivalent modulus of elasticity
BE	Binding energy	E_0	Rubbery elastic modulus
BGG	Bovine gamma-globulin	E_∞	Glassy elastic modulus
BSA	Bovine serum albumin	H_c	Central dimensionless film thickness
BSM	Bovine submaxillary mucin	H_{min}	Minimum dimensionless film thickness
CD	Circular dichroism	R'	Effective radius of curvature
DLS	Dynamic light scattering	R_a	Centre line average roughness
EHL	Elasto-hydrodynamic lubrication	R_q	Mean square roughness
HA	Hydroxyapatite	d_{50}	Median diameter
HD	Hydrodynamic	u_c	Critical entrainment speed
IDDSI	International dysphagia diet standardisation initiative	η_{eff}	Effective viscosity
LF	Lactoferrin	μ_0	Friction coefficient in the speed-independent regime
MTM	Mini traction machine	μ_{EHL}	Friction coefficient in EHL regime
MWP	Microparticulated whey protein	σ_c	Composite surface roughness

PDMS	Polydimethylsiloxane	τ_i	Interfacial shear strength
ppm	Parts per million	ΔD	Dissipation shift
QCM-D	Quartz crystal microbalance with dissipation monitoring	Δf	Frequency shift
SAXS	Small angle X-ray scattering	h	Lubricant film thickness
SE	Spectroscopic ellipsometry	η	Viscosity
SPR	Surface plasmon resonance	E	Modulus of elasticity
SRR	Slide-to-roll ratio	F	Normal load
SUP	Supercharged polypeptides	H	Dimensionless film thickness
UHMWP	Ultra-high-molecular-weight polyethylene	R	Radius of curvature
UN	United nation	T	Time
VBL	Viscous boundary layer	U	Dimensionless speed parameter
VESM	Viscoelasticity of soft material	W	Dimensionless load parameter
XPS	X-ray photoelectron microscopy	k	Ellipticity parameter
		p	Pressure
		t	Viscoelastic relaxation time scale
		u	Entrainment speed
		δ	Indentation/deformation depth
		λ	Dimensionless film thickness parameter
		μ	Friction coefficient
		ξ	Mesh size of a hydrogel network
		σ	Surface roughness
		τ	Shear stress
		υ	Poisson ratio

2. Theories of lubrication

Classical theories of lubrication define four regimes of lubrication which are boundary, mixed, elasto-hydrodynamic (EHL) and hydrodynamic (HD). The transition between regimes has often been defined qualitatively through changes in the friction coefficient values in the form of a Stribeck curve (**Figure 2**), which presents data as a function of speed-dependent parameter that is related to the film thickness between substrates (Bongaerts, et al., 2007; Hamrock, 1994; Stribeck, 1902). In common practice, the minimum film thickness (h_{min}), in a lubricated contact, is calculated assuming smooth surfaces and hence ignoring the influence of surface texture. The surface texture principally is important when contact surfaces are in close proximity, where opposing asperities (*i.e.* peaks of the surface roughness) encounter direct contacts. Therefore, tribologists defined a dimensionless film thickness parameter (λ) which encompasses the influence of surface roughness (σ) and provides an indication of the overall degree of direct asperity contact. To incorporate the impact of surface roughness of both contact bodies (σ_1 and σ_2), a parameter has been

derived and referred to as combined or composite surface roughness (σ_c). λ is given by equation (1) (Hamrock, 1994).

$$\lambda = \frac{h_{min}}{\sigma_c} \text{ and } \sigma_c = \sqrt{\sigma_1^2 + \sigma_2^2} \quad (1)$$

In common practice, σ in equation (1) has been represented by roughness parameters including the mean square roughness (R_q) or the centre line average roughness (R_a). Tall asperities has more pronounced impact on R_q value than on R_a value. Therefore, R_q is more often used in tribology and theoretical studies as opposed to R_a, latter is commonly relevant for manufacturing of engineering parts such as quality assessment reports. A tribo-contact operating at $\lambda < 1$ (a high degree of contact expected to occur between the surfaces) and $\lambda > 10$ (the minimum possibility of surface contact) is generally attributed to boundary regime and hydrodynamic lubrication regimes respectively (Hamrock & Dowson, 1981). Upon increase of λ to values over 1, one expects to gain contribution of the fluid film to load bearing and, thereby reduction in the direct contact between surfaces. The elasto-hydrodynamic (EHL) lubrication regime exploits elastic contribution of contact bodies and occurs just before the HD regime where full-film lubrication is developed. As shown in **Figure 2** using a black solid line, the friction coefficient increases in the hydrodynamic regime as the lubricant film grows thicker.

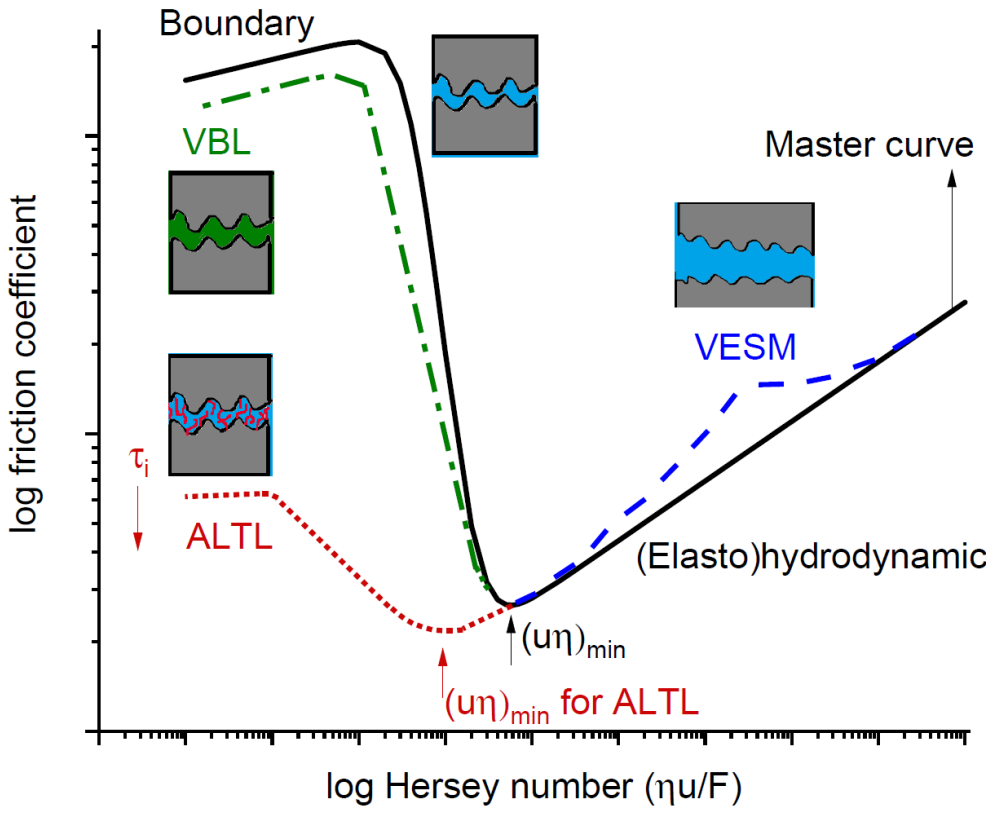


Figure 2. A typical Stribeck curve is presented in black line. The influence of viscoelasticity of soft material (VESM) on the friction coefficient of the EHL regime is shown in dashed blue line. The lubricant retention at the contact interface through a viscous boundary layer (VBL) of a viscous lubricant is shown in dash-dotted green line. Adsorbed lubricious thin layers (ALTL) has considerable influence on the lubrication performance in the mixed and boundary regime and this influence is demonstrated using a dotted red line. Friction coefficient is obtained by dividing the frictional (lateral) forces by the normal load. The Hersey number ($^{\eta u}/_{F}$) is a dimensionless parameter initially designed for journal bearing application. The Hersey number is the product of the lubricant viscosity (η), entrainment speed (u) and the contact load per unit of contact radius (F). Therefore, the Hersey number embraces the influence of fluid viscosity and the contact load (provided the film thickness remains constant by change of load) and displays the influence of speed change on frictional forces**.**

In lubrication theory when the contacting surfaces are fully separated by a lubricant film (*i.e.* full-film lubrication), further classifications are conducted to illuminate the influence of material elasticity and viscous behaviour of the lubricant on the lubricant film thickness. These are addressed in the following section.

Isoviscous elastic lubrication regime. In the full-film lubrication, the influence of material elasticity and fluid viscosity effects demarcate four types of full-film lubrication regimes especially observed in elliptical contacts. These are isoviscous-rigid, viscous-rigid, elastohydrodynamic of materials with low elastic-modulus (*i.e.* isoviscous-elastic) and

viscous-elastic (Hamrock & Dowson, 1978b). These regimes are distinguished based on the influence of contact pressure on the magnitude of solid elasticity and lubricant viscosity (Hamrock & Dowson, 1978b) with the isoviscous-elastic regime probably being the most prevalent condition in the oral tribology considering the relatively low contact pressures (30-50 kPa) and shear and elastic moduli in the oral processes (tongue's shear modulus of 2.5 kPa) (Sarkar, et al., 2019a). The compression tests on pig's tongue samples have suggested an elastic modulus of around 2.5-15 kPa (Andablo-Reyes, et al., 2020; Dresselhuis, de Hoog, Cohen Stuart, & van Aken, 2008).

The governing equations to calculate the isoviscous-elastic fluid-film thickness ($H = \frac{h}{R'}$) for soft elliptical contacts are given by equations (2) and (3) (Hamrock & Dowson, 1978a):

$$H_{min} = 7.43(1 - 0.85e^{-0.31k})U^{0.65}W^{-0.21} \quad (2)$$

$$H_c = 7.32(1 - 0.72e^{-0.28k})U^{0.64}W^{-0.22} \quad (3)$$

where, H is dimensionless film thickness, h is the film thickness, U is dimensionless speed parameter ($\frac{u\eta}{E'R'}$), W is dimensionless load parameter ($\frac{F}{E'R'^2}$) and k is the ellipticity parameter, latter is the ratio of the semi-major axis to the semi-minor axis of the contact ellipse. Terms u, η and F are the mean surface velocity (entrainment speed), viscosity of the fluid and the normal applied load. R' and E' are the effective radius of curvature in the direction of the fluid entrainment and the equivalent modulus of elasticity of contacting bodies respectively. R' and E' are given by equation (4):

$$\frac{1}{E'} = \frac{1}{2}\left(\frac{1 - {v_1}^2}{E_1} + \frac{1 - {v_2}^2}{E_2}\right) \; and \; \frac{1}{R'} = \frac{1}{R_1} + \frac{1}{R_2} \quad (4)$$

where, (E_1, E_2), (υ_1, υ_2) and (R_1, R_2) are the elastic moduli, Poisson's ratios and the radii of curvature respectively of the two contact bodies (1 and 2). A k value of 1 defines circular contacts and values above 2 represent contact configurations approaching line contacts of soft elastic bodies (see **Figure 3**). This indicates that the film thickness grows towards that of the line contact with an increase in the ellipticity parameter, provided that dimensionless speed, load, and material parameters (the equivalent modulus of the bodies and the pressure-viscosity coefficient of the lubricant) remained constant. k is a function of the principal radii of the contacting bodies and can be predicted using the following empirical equation (equation (5)) (Brewe & Hamrock, 1977):

$$k = 1.0339\left(\frac{R'_x}{R'_y}\right)^{0.636} \quad (5)$$

where, R'_x and R'_y denote the effective radius of curvature in x and y direction of the coordinate systems, respectively.

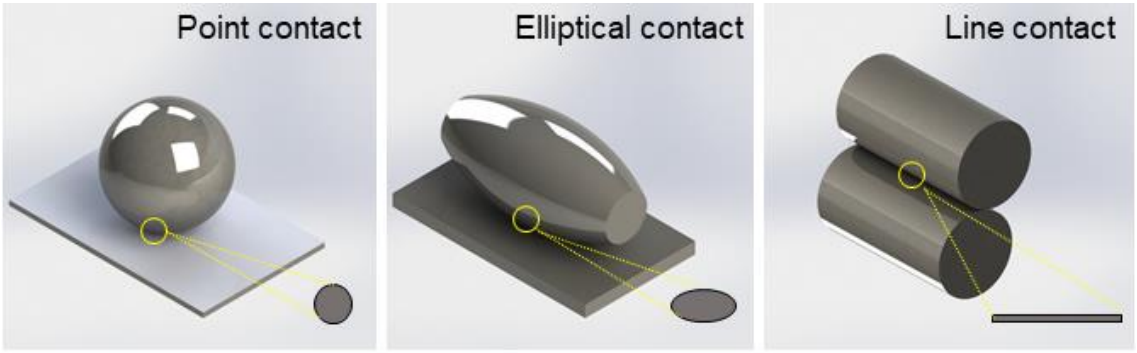


Figure 3. Illustration of contact configurations and resultant point (k=1), elliptical (k≠1 and<12) and line (k>12) contacts.

The film thickness equations can be derived by solving Reynolds equation taking into account the material compressibility and the fluid viscous effects when the entrainment occurs along the minor axis of the contact ellipse allowing the minimum film thickness to take place at the contact centre-line (Hooke, 1995). The extent of contact deformation and

entrainment direction can shift the place where the minimum film thickness takes place and hence altering the governing equations (de Vicente, Stokes, & Spikes, 2005; Hooke, 1995).

de Vicente, et al. (2005) numerically solved a 2D problem of Reynolds equation and the elasticity equation simultaneously for a fully-flooded point contact lubricated with Newtonian fluids. In solving Reynolds equation, it was assumed that the contact bodies have velocity vectors only in the direction of the lubricant entrainment and the action of the squeeze film ($12\eta\frac{\partial h}{\partial T}, T\ is\ time$) is negligible. The obtained film thickness equations for isoviscous-elastic point contact ($k = 1$) are shown in equations (6) and (7):

$$H_{min} = 2.8U^{0.66}W^{-0.22} \quad (6)$$

$$H_c = 3.3U^{0.60}W^{-0.14} \quad (7)$$

The relations showed relatively good agreement with equations (2) and (3) (Hamrock & Dowson, 1978a), except a weaker load dependence in the case of central film thickness. An experimental work, however, showed that the theoretical models may overestimate the film thickness and following equations (equations (8) and (9)) were showed the best fit for results obtained in the study (Myant, Fowell, Spikes, & Stokes, 2010):

$$H_{min} = 2.8U^{0.68}W^{-0.20} \quad (8)$$

$$H_c = 3.3U^{0.63}W^{-0.13} \quad (9)$$

Integration of viscous shear stress over the contact area of the interface yields the viscous friction force acting on each surface which then can be converted into the friction coefficient by dividing that by the normal force. Assuming the lubricant film is thin (no gradient of pressure, p, in the direction perpendicular to the lubricant entrainment), viscous

shear forces (τ) for Newtonian fluids on each contact body (*1* is the upper body and *2* is the lower body) can be calculated using equation (10):

$$\tau_1 = -\left(\frac{u_1 - u_2}{h}\right)\eta - \frac{h}{2}\frac{\partial p}{\partial x} \; and \; \tau_2 = \left(\frac{u_1 - u_2}{h}\right)\eta - \frac{h}{2}\frac{\partial p}{\partial x} \tag{10}$$

In the work by de Vicente, et al. (2005), prediction of friction, therefore, was carried out through integration of velocity-dependent (Couette flow, $\tau_{Couette} = \frac{\Delta u}{h}\eta$) and pressure-dependent (Poiseuille flow, $\tau_{Poiseuille} = \frac{h}{2}\frac{\partial p}{\partial x}$) shear stresses. The regression fitting of the data brought about equation (11), provided that the contact is immersed in a lubricant bath filling the diverging region of the contact:

$$\mu = 1.46U^{0.65}W^{-0.70} + SRR(3.8U^{0.71}W^{-0.76} + 0.96U^{0.36}W^{-0.11}) \tag{11}$$

where, SRR denotes slide-to-roll ratio between two bodies (1 and 2) which is presented in equation (12):

$$SRR = \frac{|u_1 - u_2|}{u_{entrainment}} \tag{12}$$

Care has to be taken in using this equation as to the Hertzian deformation being small enough relative to the material thickness which satisfies the assumption of bulk elastic material (de Vicente, et al., 2005). Masticatory and biting forces on the teeth are shown to be in the range of 0.05-0.1 kN and 0.2–2 kN (Sessle, 2014). Also, able-bodied individuals were observed to be capable of applying forces in the range of 13 N (Robinovitch, Hershler, & Romilly, 1991). Assuming similar masticatory forces on tongue (50 N) and load-applying capability of tongue (13 N), popcorn kernels with a diameter of around 5 mm and an elastic

modulus of 325 MPa (Williams, Wright, Truong, Daubert, & Vinyard, 2005) can induce indentation depths (δ) of around 7.0 and 2.8 cm based on the Hertzian contact mechanics for two hemispheres (equation (13)):

$$\delta = \left(\frac{9F^2}{8R'E'^2}\right)^{1/3} \quad (13)$$

The extent of deformation on the tongue surface in those cases is considerable compared to the tongue thickness and in this case the tongue can be considered as a soft layer on top of the floor of the mouth. Relevant equations for film thickness measurement of soft layered rigid materials can be obtained from a work by Dowson and Yao (1994). However, the movement of the tongue and the soft nature of the sublingual salivary glands and muscles of the tongue (genioglossus or hyoglossus) make the problem further complex. This suggests that the developed equations for soft-layered rigid bodies might not be applicable. Moreover, tongue similar to articular cartilage exhibits anisotropic, time-dependent and nonlinear mechanical characteristics and the values of between 0.4-9 kPa reported for its shear modules (Wang, He, Wu, & Zhang, 2018) suggests that the modulus for tongue can vary depending on the loading rate and depth. Therefore, a strain-stiffening property can be envisaged for tongue which mitigates large deformation under compression. The "*toe stage*" that is the initial nonlinear behaviour of soft tissues has been experimentally observed in tensile tests (Wang, et al., 2018).

Further, it is vital to perceive the methods used by state of the art tribometers to generate processed data (*e.g.* friction coefficient). For example, Mini Traction Machine (MTM, PCS Instruments, UK) often collects two sets of data at a specific entrainment speed, where the body *a* rotates faster as compared to the body *b* during the first set and slower during the second set. This method has been implemented to rule out the influence of zero offset reading by the force transducer. The difference between these two sets of data has

been used to calculate the friction coefficient (de Vicente, et al., 2005). The Poiseuille flow contribution to the viscous shear forces is independent of the sliding direction and hence this method leads to the Poiseuille flow being omitted from the results. In this case, one should use only the second term in the de-Vicente equation (*i.e.* $SRR(3.8U^{0.71}W^{-0.76} + 0.96U^{0.36}W^{-0.11})$) for prediction of the friction coefficient. In general, assumptions have been made to derive equations and those have to be thoroughly considered for the application. For example, in solving Reynolds equations often inertia and gravity forces have been assumed to be negligible and slip at the boundary of the lubricant and the surfaces and non-laminar flow have been regarded as unlikely/trivial which are relevant in most cases.

Viscoelasticity of the soft body including influence on the EHL. Prediction of the film thickness and friction coefficient can become more intricate when the contribution of viscoelasticity of the contact bodies plays a considerable role. The viscoelasticity of the material potentially leads to asymmetric pressure distribution (Hooke & Huang, 1997) and rupture/ distortion of the elasto-hydrodynamic lubricant film at the inlet of the contact (Hutt & Persson, 2016). Therefore, shifting the minimum film thickness to contact inlet, whereas one would usually expect the minimum film thickness to happen at the flow outlet. As shown in **Figure 2** using a blue dashed line (VESM, a theoretical prediction), this eventually voids the linear correlation between the friction coefficient and entrainment speed in log-log scale. Upon increase in a dimensionless speed parameter *(i.e.* $^{ut}/_{R}$, where t is the characteristic viscoelastic relaxation time of the material) in a rigid-ball on viscoelastic disc configuration, the pressure distributed in a asymmetric shape with a spike in the contact inlet propelling the minimum film thickness to the lubricant inlet tend to risk a lubricant starvation (Putignano & Dini, 2017). This is accompanied with the incomplete relaxation of the material at the outlet region and stiffening effect of lubricant flow leading to a deviation from the Hertzian circular

contact zone and the linear correlation of friction with the logarithm of the speed (see **Figure 2**).

The study by Putignano and Dini (2017) exhibited a critical entrainment speed (u_c) for a rigid ball rolling against a soft disc configuration above which visco-elasto-hydrodynamic regime may be observed, as given by equation (14).

$$u_c = \frac{R}{\pi t}\sqrt{\frac{E_\infty}{E_0}} \tag{14}$$

E_∞ and E_0 are glassy and rubbery elastic moduli of the material. Depending on the load and material properties (*e.g.* relatively larger t values), if it transpired that u_c falls into the hydrodynamic region, one would expect to see a classical transition of EHL to hydrodynamic regime. On the other hand, the visco-elasto-hydrodynamic regime occurs when the material deformation at u_c is comparable to the lubricant film thickness, that is $\frac{h_{Hydrostatic}}{\delta} \approx 1\ or\ smaller$,where $h_{Hydrostatic} \propto R^3(\frac{\eta u_c}{F})^2$.

In a work by Wang, et al. (2018), the characteristic relaxation time ($t\ in\ e^{-\frac{T}{t}}$) for tongue tissue samples from boars appeared to be in the range of 80-300 s in tensile tests at a strain rate of $\sim 3 \times 10^{-5}\ s^{-1}$. The relaxation characteristic time constant observed by Wang, et al. (2018) is large and probably resulted from extremely low strain rate and relaxation modelling with a single-component exponential decay function (Abbott, Lowy, & Russell, 1957; Van Loocke, Lyons, & Simms, 2008). Unfortunately, literature lacks a comprehensive understanding of stress relaxation phenomena in human tongue. However, stress relaxation results for porcine muscle tissue have showed three characteristic relaxation time constants of sub second (10^{-5}), few seconds and order of 100 seconds (Fitzgerald, Bootsma, Berberich, & Sparks, 2015). Assuming similar relaxation time

393 constants for tongue, $\frac{E_\infty}{E_0}$ ratio of 10-100 and the tribo-contact between popcorn kernels and
394 tongue (see previous section, *i.e.* Isoviscous elastic lubrication regime), in short-lived
395 contacts (well below a second), the viscoelastic contribution seems to be unlikely as the
396 critical entrainment speed will be in the order of few hundred meters per second. However,
397 if it happened that the contact time satisfies the second stress relaxation incident (few
398 seconds), the critical speed will be in the order of few mm/s and hence deviation from
399 isoviscous elastic lubrication regime can be expected. The viscoelastic contribution can be
400 more pronounced in the tongue tip and soft palate where the greatest viscoelasticity has
401 been observed (Wang, et al., 2018). Further research works are essential to determine
402 strain-stiffening and stress relaxation behaviour (viscoelasticity/poro-viscoelasticity) of
403 tongue and to elucidate characteristic parameters of viscoelasticity (time constants). This
404 information will shed light on occasions where consideration of tongue viscoelasticity is
405 required, for example friction behaviour in the hydrodynamic lubrication regime presented
406 in **Figure 2**.
407 The above statements indicate that the hysteresis losses influence the friction
408 coefficient of the full-film regime and therefore, different friction values are expected to be
409 observed for example for a rigid-ball on soft-disc (*e.g.* popcorn kernels on tongue)
410 configuration as compared to a soft-ball on soft-disc (*e.g.* meatball on tongue) configuration.
411 This implies that elimination of hysteresis induced frictional forces should yield interfacial
412 friction forces independent of the contact configuration. This hypothesis was recently
413 implemented (Sadowski & Stupkiewicz, 2019) through estimation of frictional forces using
414 Persson's equation for rolling friction of viscoelastic materials (Persson, 2010) and
415 performing correction for the hysteresis losses. The experimental results following the
416 correction showed that the full-film interfacial friction coefficients for different configurations
417 (soft-on-hard, hard-on-soft and soft-on-soft) follow a single regression equation (equation
418 15) (Sadowski & Stupkiewicz, 2019):

419

Friction coefficient in EHL lubrication regime, $\mu_{EHL} = 6.05(\frac{U}{W})^{0.547}$ (15)

420

421 The logarithm of the interfacial friction coefficient increased linearly with the logarithm
422 of the Hersey number ($\mathfrak{n}u/F$). However, as one can expect, the pressure distribution and
423 film thickness profile, where hysteresis forces play a role, alter considerably from one to the
424 other configuration and this is shown by Putignano (2020) in **Figure 2** (blue dashed line,
425 VESM). It should be noted the Persson's model (Persson, 2010) and the work by Sadowski
426 and Stupkiewicz (2019) assume negligible influence of thermal effects which impact film
427 thickness and friction (usually at high rolling/sliding speeds). This seems to be a valid
428 assumption in oral tribology considering low entrainment speeds and vascular nature of oral
429 surfaces.

430

431 **Viscoelastic effects within mixed/ boundary regimes**: The pioneering work by Bongaerts
432 et al. (2007a) showed that an empirical model can provide a 'master' curve (shown in **Figure**
433 **2**, black solid line) that embraces the influence of fluid viscosity based on the Sommerfeld
434 number (Hersey number enclosing design parameters). This approach has proved vital in
435 interpreting the fiction associated with rheologically and structurally complex food systems.
436 However, the master curve does not decipher some viscosity-dependent peculiarities in the
437 lubrication of soft viscoelastic surfaces especially in the boundary-mixed regimes (Selway,
438 Chan, & Stokes, 2017; Yakubov, McColl, Bongaerts, & Ramsden, 2009), and anticipated
439 from a theoretical model of Scaraggi and Persson (2014). For example, a work by one of
440 the co-authors showed that the boundary friction coefficients for lubricants composed of a
441 mixture of glycerol and water at different ratios were not identical (Selway, et al., 2017). This
442 was attributed to the decaying influence of viscosity on de-wetting and squeeze-out
443 dynamics of the lubricant molecules at the contact interface. This brings about a lower

friction coefficient in the mixed and boundary lubrication regimes as presented in **Figure 2** using green dash-dotted line (VBL).

Further, viscoelastic deformation of roughness on soft surfaces (tongue with papillae on top resembles an engineering soft material with protruding features classified as roughness) results in changes in the frictional behaviour in the boundary and transition to the mixed lubrication regimes. This is experimentally shown by the author where time-dependant deformation of asperities on a rough viscoelastic elastomer exacerbate the friction prediction in mixed lubrication regime (Selway, et al., 2017). This evidences that the soft material hysteresis is of paramount importance where material deformation influences lubricant flow and energy dissipation in regimes pertaining to the oral tribology (boundary to EHL).

Influence of adsorbed lubricious thin layers/moieties. Adsorbed molecules play important role in boundary friction of surfaces (see **Figure 2**, the dotted red line). In literature, it has generally been suggested that greater degrees of surface coverages of self-assembly monolayers bring about lower friction coefficients in macro-scale tribo tests (Fry, Moody, Spikes, & Wong, 2020) which were complemented by nanoscale experiments where atomic force microscopy (AFM) tips were around 20 nm (and 47 nm in post-worn condition) (Brukman, Oncins, Dunbar, Boardman, & Carpick, 2006). This correlation, however, was contradicted where a sharper tip of ~15 nm was used (Nalam, Pham, Castillo, & Espinosa-Marzal, 2019). This is attributed to the influence of tip geometry and plowing mechanisms generating defects in the adsorbed monolayer chains (Summers, Iacovella, Cummings, & McCabe, 2017).

In the macroscale tribology, however, the influence of adsorbed thin films (*e.g.* saliva) is known to impact frictional forces in boundary and mixed regimes (Krop, et al., 2019; Macakova, Yakubov, Plunkett, & Stokes, 2010, 2011; Sarkar, et al., 2019a; Sarkar, et al.,

2017a; Yakubov, et al., 2009). Stokes, et al. (2011) showed that adsorption of polysaccharides from their aqueous solution on to elastomer surfaces can reduce the interfacial shear strength ($\tau_i = \frac{Friction\ force}{Contact\ area}$) between the contact bodies and therefore reduce the frictional forces at the interface. This is presented in **Figure 2** where adsorbed thin layers shown in red dotted line (ALTL) reduce the friction coefficient of the boundary and mixed lubrication regimes. Moreover, the adsorbed thin layers may influence $u\eta_{eff}$ (*i.e.* the product of entrainment speed and an effective viscosity) value where the transition between mixed and hydrodynamic regimes happens (that is, $(u\eta)_{min}$). As can be seen in **Figure 2** (ALTL), the transition occurred at lower values of $(u\eta)_{min}$ when there is an adsorbed thin layer on the surface which was suggest to mask the effect of surface roughness (Stokes, et al., 2011).

Methodological approaches to conduct tribotests. Overall, this section highlights the importance of lubrication theories and how they can be used to understand the lubrication regimes and transitions between regimes including the importance of adsorption, when testing tribology of different food colloids. In tribology science, wear and frictional behaviour of tribopairs which the contacts undergo are of paramount importance. In tribology of soft materials, extensive attempts have been made to understand the frictional properties of materials or the lubricant in between soft surfaces. This is partly due to the fact that soft visco/poroelastic materials can dissipate the frictional and normal forces (through viscous or (poro)elastic deformation) and hence resist against wear damages. This is also accompanied with relatively lower contact pressures (primarily in the order of kPa to a few MPa) that they are subjected as compared to contact pressures in tribo contacts of hard- *e.g.* metallic/ceramic- materials (in the range of few tens of MPa to few GPa). The contact pressure has a noticeable impact on frictional behaviour of tribo-contacts. Therefore, when planning to perform tribological investigation of a material or lubricant, one needs to

familiarise themselves with the Hertzian contact mechanics and select a normal load which would yield contact pressures occurring in real conditions. Contact pressure is a function of normal load (F), contact configuration (R) and material properties $(E\ and\ v)$. Contact configuration (elliptical, line or circular contacts) impacts the distribution of contact pressure along the contact area and hence, the friction. As briefly described in this section, roughness and surface topography parameters influence friction results obtained with defined contact conditions. A recently developed methodology to produce materials containing papillae-shaped features (Andablo-Reyes, et al., 2020) using soft elastomeric material has progressed the tribology of oral surface to acknowledge the influence of roughness on frictional properties of food products.

To achieve robust results, it is desirable to imitate the governing real conditions which is being investigated and implement them into the designed tribotests. Alongside contact conditions and material properties of the surfaces, it is important to consider other influencing factors. For example, an exemplary design of tribo experiments takes into account the existence or absence of pre-adsorbed saliva film in a dynamic set-up (Fan, et al., 2021). Finally cleaning procedures of materials and tribotester for tribo experiments where adsorption of moieties from lubricant onto the surfaces takes place, are crucial, as residuals of adsorbed moieties would influence frictional results.

3. Applications of oral tribology in food colloid research

From a historical perspective, oral friction has attracted attention in food colloids community due to its key role in predicting some of the textural attributes such as smoothness and creaminess (Kokini, 1987; Kokini & Cussler, 1983), which could not be understood in physical terms using rheological characterization alone. Besides achieving sensory prediction (Pradal & Stokes, 2016; Prakash, et al., 2013; Sarkar & Krop, 2019), oral tribology has gained significant momentum to address a range of unresolved challenges in food

colloid science over the last few decades. We have identified four key challenge areas *i.e.* 1) designing healthier colloids, 2) detecting food adulteration, 3) tailoring food for vulnerable population, and 4) contributing to achieving food sustainability; where oral tribology has either already acted as a catalyst to provide the underlying mechanism to some of the questions or is expected to provide new insights in the coming years. Particularly area 1 is most evolved in terms of tribological outputs as compared to 2-4 (see **Table 2** for a summary of recent studies). We summarize the recent findings in each of these challenge areas followed by raising important questions that needs to be resolved using trbological understanding.

Table 2. Summary of use of oral tribology in key food colloid applications in the last 10 years.

Hydrocolloids/ colloidal systems	Designing healthier colloids	Detecting food adulteration	Tailoring food for vulnerable population	Contributing to achieving food sustainability	References
Polysaccharides incl. dietary fibres	✓		✓		(Kieserling, Vu, Drusch, & Schalow, 2019; Kiumarsi, et al., 2019; Nguyen, Kravchuk, Bhandari, & Prakash, 2017; Stokes, et al., 2011; Torres, et al., 2019; Vieira, et al., 2020; Xu & Stokes, 2020; Zhu, Bhandari, Pang, Liu, & Prakash, 2019a; Zhu, Bhandari, & Prakash, 2018, 2019b, 2020)
Proteins	✓			✓	(Fan, Shewan, Smyth, Yakubov, & Stokes, 2021; Laiho, Williams, Poelman, Appelqvist, & Logan, 2017; Zembyla, et al., 2021; Zhu, et al., 2019b, 2020)
Emulsions	✓				(Douaire, et al., 2014; Oppermann,

					et al., 2017; Upadhyay & Chen, 2019)
Oil bodies	✓			✓	(Yang, et al., 2020)
Starch ghost granules	✓			✓	(Zhang, et al., 2017)
Hydrogels	✓		✓		(Hu, Andablo-Reyes, Soltanahmadi, & Sarkar, 2020; Krop, et al., 2019; Stribiţcaia, et al., 2020b)
Fluid gels	✓				(Fernández Farrés & Norton, 2015; Garrec & Norton, 2013; Hamilton & Norton, 2016)
Microgels	✓				(Andablo-Reyes, et al., 2019; Hu, et al., 2020; Sarkar, et al., 2017a; Torres, et al., 2018)
Microparticulated proteins	✓				(Liu, et al., 2016; Olivares, Shahrivar, & de Vicente, 2019)
Melamine in milk systems		✓			(Liu, Hu, Zhong, & Xu, 2018)

Designing healthier colloids. Due to continuous rise in obesity and overweight population around the globe and associated morbidity and mortality consequences, there has been a continual effort in food industries and research communities to replace or reduce calorie dense fat content in food. However, such reduction or replacement of fat often comes at a price of compromised textural quality, which eventually leads to rejection of the low fat and no-fat foods by consumers and thus, refutes the overall purpose of designing low calorie products. To address this, one of the strategies used by food industries has been to match the viscosity of the low fat food with the control *i.e.* high fat counterparts. Although emulating viscosity by using biopolymers, processing *etc.* have helped to match the so-called sensory thickness of the products, but it still did not have the optimized textural properties to allow matching the mouthfeel to that of the full-fat counterparts allowing the prominence of tribology in recent investigations.

Particularly two previous studies from separate groups (Laguna, et al., 2017a; Selway & Stokes, 2013) have shown that low-fat semi-solid colloidal foods such as yoghurts, custards, cream cheeses having similar viscosities to the full fat counterparts have an order-of-magnitude higher boundary friction as compared to the full fat products and are perceived to be less creamy. As schematically shown in **Figure 4a**, these products varying significantly in fat content have similar profile of friction in the full film lubrication regimes, which is expected owing to their similar viscosity profiles (Laguna, et al., 2017a; Selway & Stokes, 2013). However, the speed-independent frictional coefficient is significantly lower in the full fat products **(Figure 4a**) due to the likely formation of a coalesced film of fat (either surface shear-induced or saliva induced) (Dresselhuis, de Hoog, Cohen Stuart, Vingerhoeds, & van Aken, 2008; Torres, et al., 2018) that forms a load bearing lubricating film at the interface unlike the low/ no-fat products.

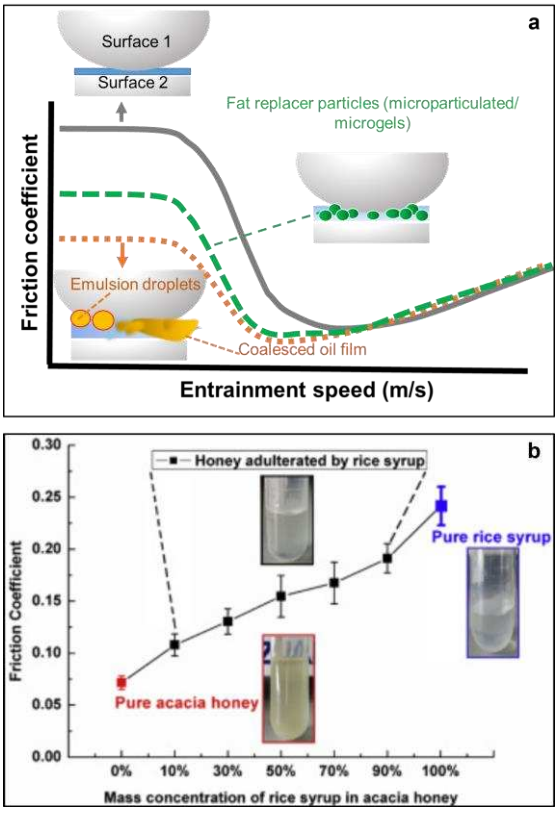


Figure 4. Examples of application of oral tribology. **a.** Schematic illustration of friction curves of fat (orange dotted line) versus non-fat iso-viscous food products (gray solid line) (Laguna, et al., 2017a; Selway & Stokes, 2013) and replacement of fat in model foods (green dashed line) using microparticulated proteins (Liu, et al., 2016; Olivares, et al., 2019), proteinaceous microgels (Andablo-Reyes, et al., 2019; Sarkar, et al., 2017a) or

biopolymeric fluid gels (Fernández Farrés & Norton, 2015; Gabriele, Spyropoulos, & Norton, 2010; Garrec & Norton, 2013). **b.** Tribology used to detect rice syrup in acacia honey (Liu, Qu, Luo, Xu, & Zhong, 2019).

Applied research ideas to replace fat by addition of hydrocolloids (Nguyen, et al., 2017), changing protein types and ratios (casein: whey protein) (Laiho, et al., 2017) in low fat dairy colloids have been in the forefront of tribological research today (**Table 2**). Furthermore, a key part of the formulation strategy has been to characterize the tribological properties of fat mimetics to understand their ability to replace or reduce fat droplets in foods. This has ranged from understanding frictional properties of diverse range of microstructures of emulsion-based systems [emulsion microgel (Torres, et al., 2018), double emulsions (Oppermann, et al., 2017), natural oil bodies (Yang, et al., 2020)] to polymeric microparticles [microparticulated proteins (Liu, et al., 2016; Olivares, et al., 2019) (biopolymeric fluid gels (Fernández Farrés & Norton, 2015; Gabriele, et al., 2010; Garrec & Norton, 2013; Hamilton & Norton, 2016), microgels (Andablo-Reyes, et al., 2019; Hu, et al., 2020; Sarkar, et al., 2017a; Torres, et al., 2018)] to microgel-reinforced hydrogels (Hu, et al., 2020) (**Table 2**). Interestingly, many of these particles have shown interesting lubrication performance (**Figure 4a**) either due to the ball-bearing mechanism of these particles emulating the lubrication performance of fat-rich emulsion droplets (Liu, et al., 2016; Sarkar, et al., 2017a) or due to entrainment and high shear viscosity properties of these particles (Andablo-Reyes, et al., 2019; Gabriele, et al., 2010) depending upon their elasticity, size, volume fraction of these particles as well as drag force of the continuum in which the particles are dispersed. A recent work (Hu, et al., 2020) has shown that reinforcing hydrogels with proteinaceous microgels can result in extremely high lubrication performance combining the properties of high viscosity of the hydrogel and adsorption properties of the microgels, such complex fluids which might find fat replacement applications in the future.

The other aspect of designing healthier colloids is to introduce dietary fibres in food systems. Fibre addition is now pretty much a standard process in solid foods such as breads

593 and meat (Angioloni & Collar, 2011; Guedes-Oliveira, Salgado, Costa-Lima, Guedes-
594 Oliveira, & Conte-Junior, 2016), where tribology has recently been shown to be a promising
595 tool with higher friction coefficients being measured for bread containing either wheat bran
596 or resistant starch fibers as compared to control or the ones containing inulin (Kiumarsi, et
597 al., 2019) (**Table 2**). Incorporation of fibres in colloidal semi-solids is still a challenge without
598 remarkable changes in texture and sensory properties. For example, recently it was shown
599 that presence of orange peel fibre was associated with increased friction coefficients in
600 yoghurt and therefore reduced sensory smoothness depending upon size (coarse
601 (d_{50} = 83 µm) or fine (d_{50} = 20 µm)) and fibre concentration (0.1-1.0%) (Kieserling, et al.,
602 2019) (**Table 2**). Although one might expect this to be linked to the particulate nature of the
603 fibre particle (*e.g.* size and stiffness) increasing the roughness in the tribological contacts,
604 authors suggested the difference in tribological properties between coarse and fine fibre
605 particle-rich yoghurts to be associated with the difference in syneresis of water from the
606 continuous network. In other words, the coarse fibre-rich yoghurt released more water due
607 to a high strain associated with increased sliding speeds during tribological measurements,
608 whilst, a reduced friction coefficient in fine fibre yoghurt was attributed to spatial embedding
609 of fine fibre particles into the casein network with strong water binding properties at high
610 strain (Kieserling, et al., 2019).

611 To sum it all up, this suggests that tribology is already getting well-acknowledged in
612 research as a quintessential mechanical tool to characterize and decipher underlying
613 mechanisms of sensory perception in fat rich and reduced fat systems as well as fibre-rich
614 systems. However, many questions remain, which challenges the regular use of tribology in
615 product design. For instance, is tribology still sensitive in understanding fat replacement
616 when the degree of replacement is subtle *i.e.* difference of fat content between the low fat
617 and fat-rich systems are in the order of 5-20% that is more common in development, which
618 can be perceived orally? Also, can tribology be used to identify differences in fat-rich

products based on fatty acid profiles such as saturated fat or trans-fat versus liquid oils, in order to design healthier colloids? One might anticipate that solid fat containing mostly saturated fatty acids offering a better surface separation between oral contact surfaces similar to that of semi-solid grease. This might lead to further reduction in friction in comparison to a low viscosity liquid oil, which might slip the contact region – this needs to be investigated in the future. For fibres, the tribological studies are thus far very limited to derive broader mechanistic understanding for a wide variety of fibre-based ingredient used in formulation design.

Another important point to raise here is that often tribology has been used in monophasic model systems. However, real foods are multiphasic where a lot of formulation and processing changes are carried out to design the low-fat or high fibre systems and thus, tribology in real food systems need more focus. Lastly, a key challenge in food science is also sugar reduction. Often such replacement require use of highly sweet non-nutritive sweeteners which are required in lower quantities than sugar and also bulking agents to contribute to the total solids. Therefore a key question that remains to be answered is whether the mouthfeel issues such as metallic after taste associated with low sugar products has an underlying tribological mechanism? We believe these questions need to be resolved with more research in the domain for tribological testing to become a regularized tool in the screening process in order to accelerate product development cycle for healthier colloids.

Detecting food adulteration. Food adulteration with cheaper and sometimes even harmful ingredients and food counterfeiting is a huge economic and public health concern in many parts of the world. And, thus, easy detection of food adulteration has attracted great deal of interests among government bodies, food industries and academic institutions. Conventionally spectroscopic, chromatographic and more recently chemometric techniques are used for detection of adulteration, which are quite time consuming and generally require

laborious sample preparation. In last two years, tribology has been used as an impressive physical diagnostic tool to detect adulteration in food (Liu, et al., 2018; Liu, et al., 2019). For instance, Liu et al. (2019) demonstrated that that liquid acacia honey shows an elastohydrodynamic lubrication whereas rice syrup shows boundary lubrication behaviour. On adulteration of honey with rice syrup, the lubrication state changes into boundary lubrication, which can be easily quantified using any tribological set-up. As shown in **Figure 4b**, the friction coefficient of natural acacia honey becomes nearly three-time larger when rice syrup is added. Similarly, tribology was used to detect melamine in milk showing that replacement of casein by melamine can increase the friction coefficient significantly (Liu, et al., 2018) (**Table 2**), however the range of melamine investigated was 20-600 ppm. According to United Nation regulations (UN, 2012), the maximum level of melamine allowed is 1 ppm and 2.5 ppm in powdered infant formula and in other foods and animal feed, respectively. Therefore it is worth noting that the lowest concentration of melamine investigated by Liu, et al. (2018) exceeded the UN regulation already by one order of magnitude. In other words, looking at the present study (Liu, et al., 2018), tribology alone might not be able to compete in terms of detection limits with other sophisticated chromatographic techniques. Nevertheless, tribology can be a potentially cheaper and simpler screening tool for *qualitative* detection of adulterants without requiring complex sample preparation, which might need further quantification using other highly sensitive techniques. Also, for other adulterants, tribology might offer frictional distinction which needs further investigation.

These case studies provide an important as well as unique application on identifying *tribological signatures* that can be used to detect food counterfeiting or adulteration. One obvious application will be to determine adulteration with one source of milk or dairy colloids when adulterated milk from other sources, let's say buffalo milk with cow milk (Trimboli, et al., 2019). If the tribological signature *i.e.* the specific profile of the tribological curves and

671 magnitude of friction at boundary and fluid film lubrication regimes are identified for a specific
672 source of milk based on the fat content or the fatty acid profile, that would be a breakthrough
673 in milk adulteration detection as tribology can be cheaper, less time consuming and would
674 require no sample preparation as opposed to most of the techniques used in literature. To
675 realize this, it is important to carry out large scale tribological analysis on a range of fatty
676 acids as well as milks from various countries and sources in the future. The case study of
677 using tribology for adulteration detection of milk can be further translated to detection of
678 other adulterants in variety of food products.
679
680 **Tailoring food for vulnerable population.** Ageing population suffers from various forms of
681 oral insufficiencies due to physiological alteration in the oral cavity such as poor dentition,
682 lower tongue pressure, lack of saliva, which often leads to impaired oral processing and
683 eventually reduced food intake and malnutrition (Belibasakis, 2018; Laguna, Hetherington,
684 Chen, Artigas, & Sarkar, 2016; Laguna, Sarkar, & Chen, 2017b; Xu, Laguna, & Sarkar,
685 2019). Dysphagia has been a commonly diagnosed eating difficulty in swallowing or moving
686 foods and liquids from the mouth to the stomach affecting approximately 8% of the global
687 population. (Cichero, et al., 2013; Marconati, et al., 2019; Zargaraan, Rastmanesh, Fadavi,
688 Zayeri, & Mohammadifar, 2013). This has been addressed by the food community largely
689 by viscosifying liquid food by adding thickeners to increase oral residence time or increasing
690 the textural complexity of foods (Cichero, et al., 2013; Cichero, et al., 2017; Laguna, et al.,
691 2016). The key to safe swallowing of food is a complex interplay between rheological
692 properties of the bolus including bolus cohesiveness, propulsive forces applied by the
693 oropharyngeal musculature and biomechanical measures utilized for protecting the airway
694 (Foegeding, Stieger, & van de Velde, 2017; Nicosia, 2007).
695 Recently, the employment of tribology in characterization of dysphagia thickeners as
696 well as thickened food have attracted research attention (Torres, et al., 2019; Vieira, et al.,

2020). The fundamental work by Stokes, et al. (2011) on tribological analysis of shear thinning polysaccharides such as pectin, xanthan gum, gellan gum, κ-carrageenan, and locus bean gum can be considered as the basis for this challenge area (**Table 2**). It was demonstrated that at the lowest sliding speeds, locust bean gum was the least lubricating while pectin reduced the friction coefficient to below 0.01. It was also highlighted that polysaccharides influenced the transitioning between the mixed and elastohydrodynamic regimes; with pectin, gellan gum, and carrageenan showing the onset of full film lubrication at significantly lower values of $u\eta_{eff}$ (entrainment speed x effective high shear rate viscosity) than xanthan gum and locust bean gum. This suggests the importance of associating rheology and tribology to distinguish the material performance of various thickeners.

In a separate study (Torres, et al., 2019), the lubrication properties of xanthan and gellan gum were compared with commercial starch-based dysphagia thickeners. The increase in boundary friction in presence of commercial starch-based thickeners was demonstrated to be associated with α-amylase-induced hydrolysis in presence of model saliva, which was not observed in gellan or xanthan gum. This study highlighted the importance of gellan gum (0.3 wt%) at a much lower concentration to provide similar lubrication property to that of xanthan gum (1.0 wt%). In addition to studying thickener solutions on their own, the tribological performances of thickener solutions (flax seed gum, xanthan gum, pregelatinized starch as well as commercial starch or xanthan-based thickeners) have been investigated when incorporated in different food matrices (water, soy juice and skim milk) (Vieira, et al., 2020). It was interesting to note that although the lubrication performance of a particular matrix was dependent on the type and concentration of the thickeners with xanthan gum showing better reduction in friction as compared to starches. But more importantly, food matrices had a crucial role to play such that milk matrix presented the highest lubrication performance as compared to water and soy juice

722 irrespective of the thickener types, highlighting the importance of studying tribology of simple
723 thickeners as well as thickeners dispersed in real-life food matrices

724 These limited yet important studies show a good starting point of using tribology in
725 addition to rheology for dysphagia management. One particular area that needs attention in
726 the future is to study bolus tribology. The role of bolus rheology for safe swallowing is well-
727 investigated (Chen & Lolivret, 2011; Ishihara, Nakauma, Funami, Odake, & Nishinari, 2011),
728 but the contribution, if any, of tribology of bolus for swallowing remains to be investigated.
729 Bolus tribology using model "*in vitro* masticated boli" *i.e.* food hydrogels + model saliva has
730 surfaced recently (Fuhrmann, et al., 2020; Krop, et al., 2019; Stribiţcaia, et al., 2020b) but
731 tribological analysis of real boli and correlation with swallowing time would be an obvious
732 next step. An important milestone in this challenge area will be the incorporation of
733 tribological parameters and/or lubrication terminologies in the International Dysphagia Diet
734 Standardisation Initiative (IDDSI) framework (Cichero, et al., 2017), provided there is
735 enough evidence of role of surface-induced tribology in swallowing after decoupling the
736 rheology-dependent tribological aspects.

737

738 **Contributing to achieving food sustainability.** Food industries are going through a huge
739 shift on moving towards use of plant and alternative sustainable ingredients as opposed to
740 rather more functional animal proteins, in particular plant proteins. Besides the functional
741 aspects, often such replacement of dairy proteins by lesser plant proteins can result in
742 sensorial issues such as astringency which have been often underestimated (Cosson, et al.,
743 2020). A question to raise here is that are such textural issues in plant proteins related to
744 lubrication failure? Interestingly, a significant amount of work has been done in tribology field
745 on astringency of polyphenol-rich wine, tea and coffee and the role of friction in quantifying
746 astringency perception is relatively well-established. In particular, in many, if not most cases,
747 astringency has been associated with high friction coefficients particularly in the low speed

748 regime which are either saliva-induced or surface-induced (Brossard, Cai, Osorio, Bordeu,
749 & Chen, 2016; Gibbins & Carpenter, 2013; Laguna & Sarkar, 2017; Rossetti, et al., 2009;
750 Wang, Olarte Mantilla, Smith, Stokes, & Smyth, 2020). In addition, it is well known that whey
751 protein induces astringency owing to charge interaction at low pH due to attractive
752 interactions between cationic whey protein and anionic mucins (Carter, Foegeding, & Drake,
753 2020). However, at neutral pH conditions that are relevant to dairy beverages, salivary
754 proteins pre-adsorbed to a substrate interact more with the casein than whey protein to
755 cause an increase in friction that directly related to sensory mouthfeel percepts (e.g.
756 smoothness) (Fan, et al., 2021).

757 To our knowledge there is only one study thus far from one of the co-author which
758 has looked at tribology of plant protein solutions. In this recent study (Zembyla, et al., 2021)
759 comparing soluble fractions of whey protein with pea protein showed that pea protein
760 solution does show lubrication performance at low concentrations (≤ 10 mg/mL) with high
761 and rapid rate of adsorption to the surface. However, at higher concentration, pea protein
762 solution showed aggregation transition from a polymer to a particle-like behaviour jamming
763 the contact resulting in lubrication failure. This might suggest indirectly that higher
764 concentration of pea protein might be detrimental to sensory perception resulting in
765 grittiness, dryness and astringency perception.

766 One might anticipate that tribology of plant proteins will become a key research area
767 in the future answering the governing principles behind astringency or other surface-induced
768 taste sensation of plant proteins. It is also worth noting that most plant proteins are
769 associated with polyphenols, which might also influence interaction with salivary mucins and
770 affect textural perception. One should also consider that most studies on astringency have
771 been carried out in liquids and not in solid composite foods, such as meat analogues where
772 use of plant proteins is garnering momentum. It is highly possible that such astringency
773 perception might not be pronounced when incorporated in a solid food or might be even

more detectable due to association with other astringent compounds. More importantly, the tribological behaviour of such solid food would depend on the degree and kinetics of oral processing and consequently on the length scale and time scales of the film formation between the oral surfaces, which needs to be examined. Although, this area appears to be at the nascent stage, we believe this will soon be populated with research due to the urgency of shifting towards plant-based diets without compromising taste and textural attributes.

4. Complementary analytical techniques for tribology

Physical and chemical analytical techniques can have a profound impact in advancing our fundamental understanding of food oral processing. While numerous multidisciplinary approaches have been taken to address tribological phenomena across variety of applications, some have provided powerful insights into the understanding of frictional properties of oral-related surfaces/ substances and some have the potential to be exploited in the future. In this section we aim to capture a few of these complementary tools that have been used as a catalyst to explain some specific frictional behaviour in food structures or allied soft materials.

Small angle X-ray scattering (SAXS). Besides measuring size using dynamic light scattering (DLS), small angle X-ray scattering (SAXS) can be a highly complementary tool to tribology to identify the transformation of food particles during oral processing, in particular to track structural changes in the range of < 100 nm length scale for colloidal dispersions (Narayanan, Wacklin, Konovalov, & Lund, 2017). Although SAXS has been recently used to analyse structure of food colloids and hydrocolloids (Adal, et al., 2017; Borah, Rappolt, Duary, & Sarkar, 2019; Gilbert, 2019; Ramel, Peyronel, & Marangoni, 2016) and also employed in combination with rheological analysis (Yu, Yakubov, Martínez-Sanz, Gilbert, &

799 Stokes, 2018) to understand conformation and intermolecular interactions in colloidal
800 systems, its use in oral tribological studies is not known to date.

801 SAXS has prompted considerable advancements in the field of soft tribology (Urueña,
802 et al., 2015). For instance, friction coefficient of polyacrylamide hydrogels (**Figure 5a**) in the
803 speed-independent regime (μ_0) has been demonstrated to inversely correlate with the mesh
804 size of the hydrogel structure (**Figure 5b**), latter obtained using SAXS (Urueña, et al., 2015).
805 On the other hand, in the speed-dependent regime, the normalized friction coefficient has
806 been found to scale with the mesh size at 1/2 power (**Figure 5c**). In machinery lubricants,
807 assessment of core diameter and aggregation of polymeric swollen nanoparticles, obtained
808 by SAXS, has led to an understanding of the boundary lubrication properties (Derry, Smith,
809 O'Hora, & Armes, 2019). This suggests that SAXS can have promising applications in
810 predicting tribological properties of food-based gels based on mesh size and degree of
811 crosslinking of the hydrocolloids as well as help understanding lubrication properties of
812 swollen food-grade microgels, which need investigation in the future.

813 Synchrotron and neutron scattering have been extensively utilised at least over the
814 last 30 years in the investigation of soft matter enabling track of (shear/ flow-induced)
815 structural changes at sub-nanometers to micron size scales (Narayanan, et al., 2017).
816 These techniques have provided a plethora of insights into the numerous areas including
817 hydrocolloids, macromolecules, where rheology and flow (Eberle and Porcar, 2012) are
818 relevant. Recently, an *in situ* shear cell with accessible scattering planes has been
819 developed to perform X-ray and neutron scattering experiments on cellulose dispersions,
820 fat crystals, caseinates in order to gain powerful insights on shear-induced changes in food
821 nanostructure (Velichko, et al., 2019). This shear cell is currently developed for “1–2 plane
822 geometry” and can be modified into cone–plate and plate–plate rheological set ups enabling
823 scattering in 1–3 plane. Nevertheless, one might anticipate that a breakthrough in this field
824 can be development of a setup that could allow probing the structural changes of bolus and

thin surface-adsorbed films (*e.g.* salivary films) under tribo-shear where parameters including normal load (contact pressure), interactions between food and surfaces and surface texture dominate the structural changes. Such hitherto unexplored approach would allow gaining fundamental knowledge on real-time data on relationship between dynamics of shear-induced nanostructural changes and frictional dissipation.

Quartz crystal microbalance with dissipation monitoring (QCM-D). Quartz crystal microbalance with dissipation monitoring (QCM-D) provides information on the adsorption kinetics and quantity (*via* measuring frequency shift), and viscous characteristics (dissipation) of surface reactive substances in a hydrated state. The interplay between the properties of adsorbed film at the interface and lubrication behaviour has been of paramount importance in tribo-contacts influencing friction, wear, and corrosion performance of the tribo-pairs.

In the last decade, QCM-D has been employed extensively in bio-relevant research works (Glumac, Ritzoulis, & Chen, 2019; Hu, et al., 2020; Macakova, et al., 2010; Marczynski, et al., 2020; Song, Winkeljann, & Lieleg, 2019; Xu, et al., 2020) especially in skin (Farias, Hsiao, & Khan, 2020), articulating joints (Majd, et al., 2014; Morgese, Cavalli, Müller, Zenobi-Wong, & Benetti, 2017; Parkes, Myant, Cann, & Wong, 2015) and food science (Pradal, Yakubov, Williams, McGuckin, & Stokes, 2019; Stokes, et al., 2011; Wan, et al., 2020b; Wang, et al., 2020; Zembyla, et al., 2021). In many studies, where the relevant tissues are hydrophobic in nature, the microbalance sensors have been coated by PDMS (Farias, et al., 2020; Pradal, et al., 2019; Song, et al., 2019; Stokes, et al., 2011; Xu, et al., 2020; Zembyla, et al., 2021) usually using spin coater instruments.

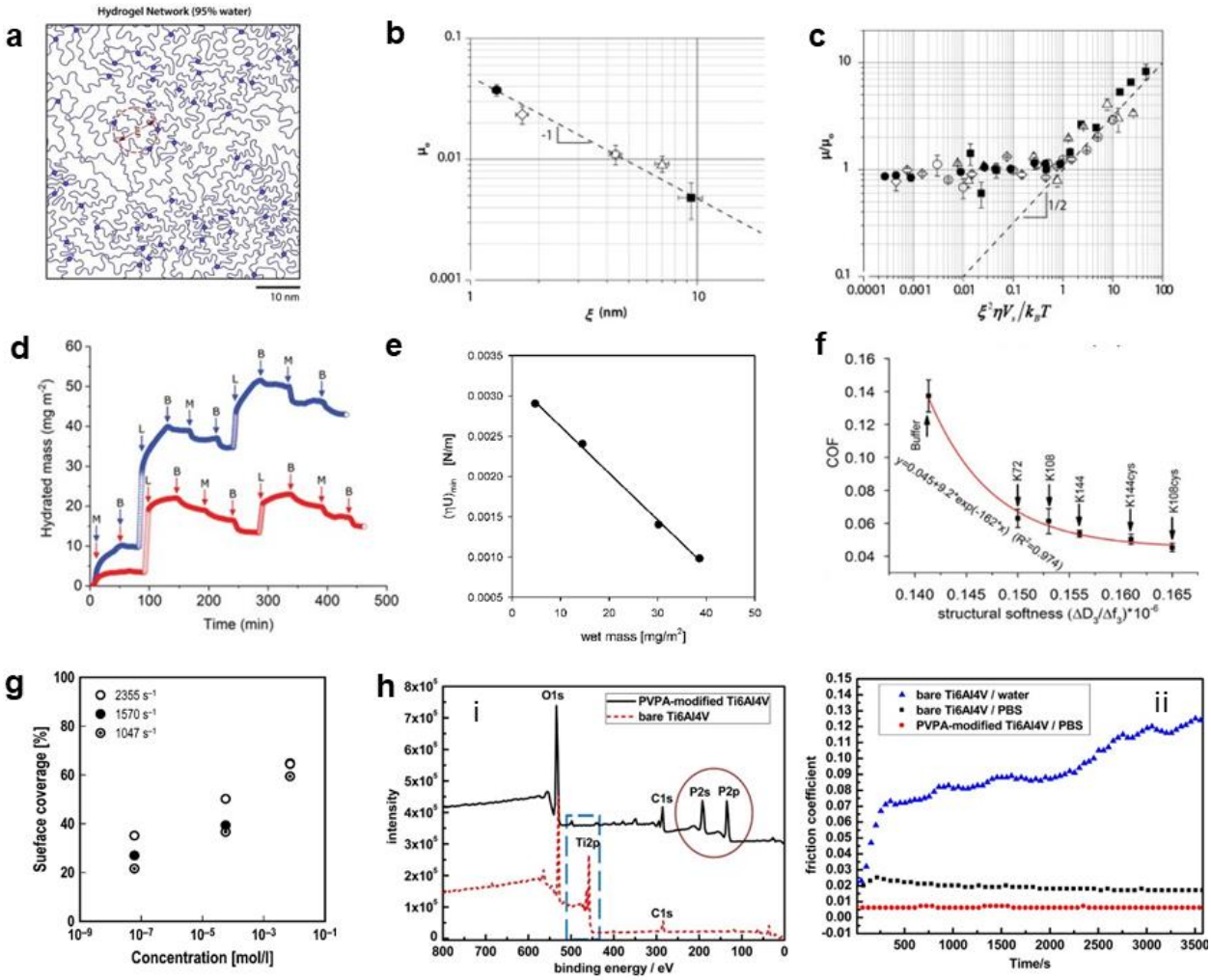


Figure 5. a) Illustration of a water-diluted polymer network (hydrogel) with a mesh size (ξ) of approximately 10 nm (Urueña, et al., 2015). b) Friction characteristics of the hydrogel in the speed-independent regime (μ_0) was inversely correlated with mesh size data obtained by SAXS (Urueña, et al., 2015). c) The normalized friction coefficient in the speed-dependant regime scales with the mesh size to a power of two (Urueña, et al., 2015). d) Measurements of adsorption of salivary proteins onto hydrophobic PDMS surfaces using QCM-D at two ionic concentrations of NaCl. The graph demonstrates incremental adsorption of hydrated mass of trilayer of BSM/LF/BSM (Xu, et al., 2020). e) The correlation between viscosity-normalised entrainment speed at the minimum friction coefficient in the Stribeck curve and mass (function of thickness) of the adsorbed film obtained by QCM-D. The graph shows that adsorbed films with thicknesses in the order of the surface roughness shift the onset of the full-film formation to lower viscosity normalised entrainment speeds (Stokes, et al., 2011). f) The correlation between structural softness (viscoelasticity) and the friction coefficient of salivary films after treatment with different recombinant supercharged polypeptides (SUP) and cysteine modified SUPs. A first-order kinetic model showed that the friction coefficient exponentially decayed with the increased structural softness as a result of SUP treatments (Wan, et al., 2020b). g) The surface coverage of adsorbed stearic acid layers in oil lubricated tribo-contacts obtained from SPR measurements as a function of shear stress and stearic acid concertation. The graph shows the higher concentrations of stearic acid and shear rates lead to greater surface mass (Maegawa, Koseki, Itoigawa, & Nakamura, 2016). h) The appearance of phosphorus signals (P 2s and P 2p) in the XPS spectrum (i) presented in black-line confirms grafting of poly(vinylphosphonic acid) polymeric chains onto Ti6Al4V surfaces. Shown in the right panel (*i.e.* ii) using red dotted-line, the grafting led to decreased friction coefficient (Zhang, Liu, Wen, & Wang, 2014).

For example, Xu, et al. (2020) recently designed a layered architecture of two salivary proteins, that were bovine submaxillary mucin (BSM) and lactoferrin (LF), formed through

electrostatic interactions between the charged proteins. The binary layered system posed similar lubrication and adsorption behaviour to those of human saliva (Xu, et al., 2020). QCM-D data demonstrated an accumulative behaviour of adsorption, where the multiple feeding of two-step injection of BSM followed by LF yielded a self-assembled bilayer, leading to a stepwise increase in the hydrated mass (see **Figure 5d**). There was a relationship between QCM-D data and lubrication such that the lubrication behaviour of the $(BSM/LF/BSM)_n$ multilayers (where n = number of layers) at macro to nanoscale resembled that of human saliva. The molecular architecture resulting in a lubricous mesh at the surface quantified using QCM-D facilitated hydration and viscous lubrication mechanism with possibility of energy dissipation- a structure similar to salivary pellicle comprising of an anchoring sublayer and a hydrated top layer at the interface (Xu, et al., 2020).

QCM-D can be useful to predict the behaviour of protein films in real contacts, which can offer new insights to protein tribology. Protein film formation from solutions of bovine serum albumin (BSA), bovine gamma-globulin (BGG), and their mixture were studied in static condition using QCM-D and in tribo-shear contacts using optical interferometry. The addition of BSA to the BGG solution was shown to decay the formation of BGG films. The results showed that the film thickness observed with QCM-D was different to that measured by interferometry in the tribo-shear condition before and during the rolling contact. However, the protein solution which engendered a greater adsorption mass and film formation rate in the static condition led to thicker films in the tribo-shear condition implying Vroman effect in shear conditions. The authors, therefore, postulated that the nature of the proteins in the solution has a dominant influence over the protein content in the solution (Parkes, et al., 2015).

In the area of oral tribology, lubrication properties of hydrocolloids are important, which have gained interest in recent decades. The friction coefficient of aqueous solutions of four polysaccharides were measured by a co-author of this paper (Stokes, et al., 2011).

A correlation between the film thickness of the hydrated polysaccharide films and viscosity-normalised entrainment speed at the minimum friction coefficient in the Stribeck curve (*i.e.* onset of the full-film formation) was observed (shown in **Figure 5e**). The adsorbed film was shown to accelerate the onset of the full-film lubrication by masking the roughness effect for relatively smooth elastomer surfaces. A more complex behaviour was observed where adsorbed film thickness assessed against lubrication properties. At the low speed range, thicker and less shear elastic films led to lower friction coefficients (Stokes, et al., 2011).

QCM-D has been used to examine the influence of the ionic strength on swelling (mass gain) and viscoelastic properties of a pectin layer covalently grafted onto PDMS surfaces through silanisation (Pradal, et al., 2019). The increase in NaCl concentration (from 1 to 100 mM) was shown to induce a thicker pectin layer and the extent of the change in thickness of the layer was shown to partially correlate with the decrease in the boundary friction depending on the degree of methyl-esterification. The shear elastic modulus of the pectin layers decreased abruptly after a certain concentration of the salt was added. This suggested that the polymer chain extension, as a result of osmotic pressure, contributes to the increase in the thickness of the layer (Pradal, et al., 2019). In this study, charge distribution of the pectin layer was postulated to be an influencing factor which limits the capability to elucidate the friction results wholly by QCM.

The findings by QCM-D do not always corroborate the findings for friction measurement instruments. This can be expected as QCM-D is a static system, which is different from the dynamic conditions and shear experienced by a sample under tribological stresses. For example, QCM-D results showed that the surface adsorption of proteoglycan-4 (lubricin) onto hydroxyapatite (HA) and collagen coated surfaces increased the friction coefficient, while lubricin adsorption on the combined surface of HA and collagen rendered a super low friction characteristics comparable to the friction coefficient observed for native cartilage (Majd, et al., 2014).

Other examples of complexity around exploiting QCM-D results to explain tribo results can occur through interactions between different macromolecules at the interface and through the influence of ionic strength which influence the surface chemistry/structure of the adsorbed layer. Song, et al. (2019) found that upon addition of lysozyme to purified porcine gastric mucins, the lubrication behaviour of mucin deteriorated in spite of an enhanced total adsorption evidenced by QCM-D. This lack of a strong correlation was also observed due to the influence of ionic strength (by addition of NaCl) on lubrication (Song, et al., 2019). Further, work by Macakova, et al. (2010) showed enhanced hydration and viscoelastic properties of adsorbed salivary films upon decrease of ionic strength (from a physiologically relevant value obtained through a solution of 70 mM NaCl to 1-10 mM NaCl). A further decrease to the minimum ionic strength (*i.e.* deionised water) led to an irreversible collapse of the multi-component salivary film which illuminates the importance of interactions between constituents of the salivary film. With reference to the friction results, the switch from a bulk salivary solution to NaCl solutions of different ionic strengths impaired the lubricity of the salivary film. This was more pronounced when the normal load was increased. These observations, therefore, do not offer a conclusive correlation between viscoelastic properties of film, obtained from QCM, and friction results from tribological experiments. It can be inferred that more detailed and fundamental studies are vital to fully comprehend the correlation (if any) between adsorption data from QCM and tribology results.

In recent studies (Wan, et al., 2020b; Xu, et al., 2020; Zembyla, et al., 2021), QCM was used to extract the softness criteria ($\Delta D/\Delta f$) for the salivary biofilms. The increase in the softness criteria translated into lower friction coefficients. A softer structure was attributed to higher water contents of the biofilm and hence the hydration lubrication was accounted for the enhanced lubricity. As shown in **Figure 5f**, the correlation between the softness criteria and the friction coefficient was shown to follow an exponential decay function (Wan, et al., 2020b).

951 Researchers should consider rheological evaluations in conjunction with QCM-D
952 when linking QCM-D with tribology results. For example, addition of thermo-responsive
953 microgels of poly(N-isopropylacrylamide)-graft-poly(ethylene glycol) (and in combination
954 with 1H-benzotriazoles) has been reported to reduce the friction coefficient of steel surfaces.
955 This was attributed to the film formation on surfaces evidenced by QCM-D (Liu, Wang, Zhou,
956 & Liu, 2013). However, the influence of apparent increases in the lubricant viscosity as a
957 result of addition of the microgel was not addressed. The lubricant viscosity directly
958 influences the film thickness (lubricant film thickness $\propto (\eta)^{0.68}$) and therefore, the impact of
959 change in viscosity, when the lubricant composition changes, should not be overlooked. In
960 other words, an increase in the lubricant viscosity can shift the lubrication conditions to less
961 harsh contact conditions resulting in a reduction in the friction coefficient.
962
963 **Surface plasmon resonance (SPR).** While gas detection and biological sensors developed
964 back in 1982 were among the first applications of the surface plasmon resonance (SPR)
965 (Chen & Ming, 2012), its current application spans from biomedical to food sciences and
966 can be used to investigate adsorption of proteins onto polymeric surfaces (Green, Davies,
967 Roberts, & Tendler, 1999). SPR utilises a laser light and detectors which track the SPR
968 angle (*i.e.* the incident angle at which the surface electrons of the substrate resonate). This
969 angle is dependent on the reflective index of the material above the substrate which
970 generates an angle shift. The angle shift is monitored in real time enabling SPR to provide
971 information on kinetics and thickness of the adsorption processes (Green, et al., 1999). Gold
972 has been commonly used as the coating for the prism due to its stability, but silver and
973 aluminium can also be used to generate the evanescent electromagnetic waves (Maegawa,
974 et al., 2016). In contrast to QCM-D, which is an acoustic based technique, the adsorption
975 data obtained from SPR represent the extent of dried mass of the adsorbate on the surface.
976 For example, the aforementioned study on the lubrication behaviour of polysaccharide

977 solutions (Stokes, et al., 2011) demonstrated an inverse correlation between the dry mass
978 of the polysaccharide films and their friction coefficients at the low-speed regime (*i.e.*
979 boundary to mixed) on relatively rough PDMS surfaces.

980 SPR can provide conformational information on the polymer adsorption and
981 therefore, is a useful tool to study the adsorption behaviour of salivary proteins. Navarro, et
982 al. (2020) conducted SPR using 50 and 100 nm diameter gold nanoparticles by which
983 different electromagnetic decay lengths were achieved (9 and 20 nm respectively).
984 Leveraged by detection at two different decay lengths (Navarro, Shah, & Zauscher, 2020),
985 SPR allowed for discerning a redistribution of the adsorbed polymer closer to the substrate
986 while the total mass of the layer remained constant during a phase where conformational
987 change happened.

988 The optical nature of the SPR effectuates *in situ* measurements. SPR has been used
989 against a gold sensor to monitor kinetics and evaluate the extent of tribology-induced
990 transfer films, *in situ*, from polytetrafluoroethylene, ultra-high-molecular-weight polyethylene
991 (UHMWP) and graphite solid lubricants (Krick, Hahn, & Sawyer, 2013). For instance, SPR
992 was coupled with microscopy of the wear track and confirmed the relocation of the transfer
993 film which was suggested by the decrease in the SPR reflectance (Krick, et al., 2013).
994 Another study (Maegawa, et al., 2016) investigated the adsorption kinetics of stearic acid, a
995 fatty acid used as friction modifier or corrosion inhibitor, in oil lubricated tribo-shear
996 experiments. The results showed that the static adsorption can take place over a large time
997 scale (in order of several hours). The study provided evidence that shear stresses promoted
998 the surface adsorption of stearic acid with increased shear rates leading to higher adsorption
999 mass (**Figure 5g**). The results indicated that when the surface coverage of the adsorbed
1000 layer is relatively large (> 50%), the influence of shear rate on the adsorption behaviour is
1001 sparse as compared to the influence of concentration of the fatty acid in the lubricant
1002 (Maegawa, et al., 2016) (see **Figure 5g**). These enlighten implications with reference to oral

1003 tribology, such that chewing and relative motion between tongue and palate influence
1004 parameters involved in the adsorption of saliva and macromolecules.
1005
1006 **Spectroscopic ellipsometry (SE).** Spectroscopic ellipsometry (SE) is another optical
1007 technique which estimates the film thickness based on measurement of the ratio of reflected
1008 and transmitted polarised light at an interface between materials described by Fresnel. SE
1009 is an elegant technique to measure the thickness of thin films in sub-nanometer resolution
1010 and has been widely used to decipher salivary adsorption on surfaces. Details derived by
1011 SE measurements assist in understanding tribological results. For example, SE has been
1012 used to estimate the grafting density of pectin layers grafted onto PDMS surfaces suggesting
1013 its brush-link conformation (Pradal, et al., 2019). The brush-like conformation of the grafted
1014 pectin layer was postulated to bring about lower friction coefficients as compared to
1015 adsorbed flatter pectin layers (Pradal, et al., 2019). SE can also evaluate the adsorption
1016 affinity of proteins (*i.e.* fouling) towards different surface treatments (Serrano, et al., 2013).
1017
1018 **X-ray photoelectron microscopy (XPS).** X-ray photoelectron microscopy (XPS) is a
1019 surface sensitive technique which provides information over the top 5-10 nm layer of surface
1020 films depending on the inelastic mean free path of the emitted electrons which travel through
1021 the surface. The electron trajectories are influenced by the properties of surfaces (*e.g.*
1022 atomic number) and the binding energy (BE) of signals. The chemical composition of surface
1023 films can be assessed through deconvolution of signals and contribution of each chemical
1024 bonding or element to surface films can be quantified using a sensitivity factor for each
1025 signal. A shift of binding energy in each signal imparts detailed information about the
1026 chemical composition of a specific compound.
1027 In biotribology, XPS has been used to confirm the grafting of lubricous layers. For
1028 examples, pectin bonding onto elastomer surfaces (Pradal, et al., 2019) was evidenced

through appearance of two additional peaks corresponding to acetal and carbonyl/ester-carboxylic groups. In another work, chemical bonding and stability of poly(vinylphosphonic acid) onto Ti6Al4V alloy, manifested in XPS spectra, was shown to bring about super-low friction characteristics (Zhang, et al., 2014) (see **Figure 5h**). XPS can be used to compare the grafting density and extent, for example, as a function of grafting time or concentration of monomer in a solution, or, more precisely, the ratio of the characteristic signals from the grafted substance (*e.g.* P 2p in **Figure 5h**) to the signal from the bare substrate (*e.g.* Ti 2p in **Figure 5h**) can indicate the grafting density or extent. Moreover, under certain conditions, the thickness of the grafted layer can be estimated through Ar ion or atom cluster etching. The grafting density/extent explicated by XPS can be correlated to the lubrication performance of the thin grafted layer.

Other works in this area involve pursuing XPS to assess grafting of dopamine conjugated hyaluronic acid onto polyurethane surfaces to enhance lubricity of cardiovascular catheters (Wan, Lin, Kaper, & Sharma, 2020a), grafting of 2-methacryloyloxyethyl phosphorylcholine onto the polydopamine coated surfaces to achieve anti-fouling surfaces (Asha, et al., 2019), atom transfer radical polymerization grafting of poly(N-hydroxyethylacrylamide) brushes and its crosslinking to facilitate aqueous lubrication (Zhang, et al., 2016) and grafting of cationic/anionic polyelectrolyte brushes (Han, et al., 2018). Further, adsorption processes (*e.g.* macromolecules onto surfaces of contact lenses) (Huo, Rudy, Wang, Ketelson, & Perry, 2012), and chemical reactions (*e.g.* redox reactions in preparation of silver-lignin core-shell nanoparticles) (Gan, et al., 2019) can be tracked by XPS to provide insight in to the associated lubrication performance.

Raman spectroscopy. Raman spectroscopy has been extensively used in characterisation of tribofilms from additives in oil lubricated systems (*e.g.* molybdenum dithiocarbamate) and amorphous carbon coatings. In biofilms, Raman can be used to identify the chemical

functional groups in adsorbed protein films and estimate their thickness (Zhang, Luo, Zhou, Zhang, & Huang, 2013). For example, a wear-induced change in the long-range order of the ultra-high-molecular-weight polyethylene structure was evidenced by Raman in trio-contacts relevant to motion in a prosthetic hip (Trommer, et al., 2015). In addition, oil-in-water emulsions, stabilized by a nonionic surfactant (Tween 60) was investigated by Raman spectroscopy and it was found that the volume fraction of oil was lower in the contact zone as compared to the bulk emulsion (Bongaerts, Day, Marriott, Pudney, & Williamson, 2008). Therefore, Raman spectroscopy can provide insight into tribology-induced microstructural reorientation of materials and hence can potentially be used in understanding of oral tribology processes, which to our knowledge has attracted limited attention to date.

Circular dichroism (CD). Circular dichroism (CD) spectroscopy uses circularly polarised light to provide information on optically active chiral bio-macromolecules based on the molecule's asymmetry leading to different absorption patterns depending on the direction of polarisation. Different detection techniques have been developed for CD to improve measurements spanning magnetic to liquid chromatography. Combination of CD with adsorption and lubrication data can provide detailed understanding of the lubrication behaviour of bio-macromolecules. For example, protease treatment of bovine submaxillary mucin was shown to deteriorate its low-speed lubrication properties (Madsen, Svensson, Abou Hachem, & Lee, 2015). The CD results in that work (Madsen, et al., 2015) showed almost unchanged random coil secondary structure of the treated mucin. These results suggested pivotal role of cleaved terminal un-glycosylated peptides and hydrophobic terminal domains in the lubrication behaviour of mucin.

In summary, this section encompassed a brief review of a few analytical tools that have been used in combination with tribological instrument which imparted better understanding of the frictional behaviour. However, there are numerous techniques which

have been exploited in the area of food science or tribology which are not included here; *e.g.* atomic force microscopy (AFM has been used for imaging and friction measurements (Majd, et al., 2014; Wan, et al., 2020b; Xu, et al., 2020). AFM has evidenced the disturbing influence of dietary tannins (*i.e.* epigallocatechin gallate) on friction characteristics and aggregation of whole salivary mucosal pellicle which were correlated with the tannin-induced astringency sensation (Ployon, et al., 2018). Further, some of the analytical techniques outlined above have been combined with tribometry equipment to form an integrated setup providing remarkable in situ/ real-time information under tribo-conditions such as integration with QCM (Borovsky, Garabedian, McAndrews, Wieser, & Burris, 2019), attenuated total reflection infrared (Piras, Rossi, & Spencer, 2003) and Raman (Bongaerts, et al., 2008). One might anticipate exploitation of these in-situ techniques to understand oral processes or attempts to develop new integrated setups comprising a tribo-equipment and an analysis technique valuable to investigate oral processes (*e.g.* in situ- tribo-SAXS). We envisage that this review paper, therefore, can trigger follow-up reviews or complement new method development specifically targeting complementary analytical tools used in food tribology in an integrated set-up.

5. Conclusions

Importance of oral tribology been recognized nearly four decades ago. Nevertheless, extensive research in tribology of food colloids has occurred relatively recently. This review provides a comprehensive view on lubrication theories, particularly occurring in soft contacts which are most relevant to oral surfaces that are deformable and flexible. The construction of the Stribeck curve and its variation under the changing conditions of the lubrication system (properties of the interacting surfaces and properties of the lubricant) are examined. Applications of oral (soft) tribology are still under exploration and under development, but sensory implications are no doubt core concern of most reported researches. Some

supporting analytical techniques have also been briefly summarized in this review, in the hope that readers can make the best use of such ancillary techniques for understanding the oral tribology results.

Oral tribology is still in its infancy. While new discoveries are waiting to be made, a series of serious challenges need to be addressed. Few examples of the same are given below:

- The soft surfaces in the oral environment are very unique. Despite some initial researches indicating the mechanical strength of the tongue, its dynamic firmness as a function of muscle contraction during oral processing remains largely unknown.
- Evidences show that tongue geometry and topography are highly irregular. Although there has been a significant progress in this direction on designing tongue-like papillated surface for tribometer showing the importance of random distribution of papillae on computationally studied mechanosensing (Andablo-Reyes, et al., 2020), relating the *in vitro* tribological data to real time *in vivo* sensory data remains as a challenge.
- The lubricating film in the oral environment could be highly dynamic. This is not only reflected by the changing thickness of the film, but more on the compositional level and mechanical properties of the film due to the continuous secretion of fresh saliva from different salivary glands, in flux of food, food residues and the instant food-saliva interactions.
- Huge disparities among human individuals (*e.g.* oral topography, saliva compositions, oral operational conditions, pattern of tongue movement and speed, *etc.*) are obvious and evident. Thus, reflecting such a high disparity in *in vitro* experimental design is a necessary undertaking.

Of course, challenges above listed are not exclusive but just to serve examples of the challenges of oral tribology domain that need consideration in the future. We hope that this

review will lead to more discussion and experimental research on this subject and such challenges.

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Conflict of Interests

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