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Relating rheology and tribology of commercial dairy colloids to sensory perception

Laura Laguna,[a] Grace Farrell,[a] Michael Bryant,[b] Ardian Morina[b] and Anwesha Sarkar*[a]

This study aims to investigate the relationship between rheological and tribological properties of commercial full fat and fat-free/low fat versions of liquid and soft solid colloidal systems (milk, yoghurt, soft cream cheese) with their sensory properties. Oscillatory measurements (strain, frequency), flow curves and tribological measurements (lubrication behaviour using Stribeck analysis) were conducted. Oral condition was mimicked using artificial saliva at 37 ○C. Discrimination test was conducted by 63 untrained consumers, followed by a qualitative questionnaire. Consumers significantly discriminated the fat-free/low fat from the full fat versions ($p < 0.01$) in all product classes, with most common verbatim used being "creamy", "sweet" for the full fat *versus* "watery", "sour" for the fat-free samples. Flow behaviour of both versions of milk showed overlapping trends with no significant differences identified both in absence and presence of saliva ($p > 0.05$). Full fat and fat free yoghurts had similar yielding behaviour and elastic modulus (G'), even in simulated oral conditions. However, in case of soft cream cheese, the full fat version had a moderately higher G' than the low fat counterpart. Even in presence of artificial saliva, there was slight but significant difference in viscoelasticity between the cream cheese variants depending on fat content ($p < 0.05$). Stribeck curve analyses showed that at lower entrainment velocities (1–100 mm s^{-1}), both full fat yoghurt and soft cream cheese exhibited a significantly lower traction coefficient when compared to fat-free/low fat versions ($p < 0.05$), which might be attributed to the lubricating effect of the coalesced fat droplets. Surprisingly, whole and skim milks showed no significant difference in traction coefficients irrespective of the entrainment speeds ($p > 0.05$). Results suggest that sensory distinction between fat-free and full fat versions, particularly in semi-solid systems could be better predicted by lubrication data as compared to bulk rheology.

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1. Introduction

The incidence of obesity is increasing at an alarming rate in the UK and worldwide. Obesity (BMI ≥30 kg m^{-2}) can be characterised by a positive energy balance, when the caloric intake exceeds energy expenditure.[1] According to the World Health Organization report in 2015,[2] more than 1.9 billion adults are overweight worldwide, and 600 million of them are obese; which equates to 13% of the world's adult population suffering from over-nutrition. Furthermore, childhood obesity (aged 0–5 years) is one of the most serious global public health challenges, with an increase of 24% in last 23 years. Excessive adiposity is related to other life threatening illnesses such as cardiovascular diseases, type 2 diabetes and some cancers.

These food-linked diseases pose considerable challenges to food industries for reformulation of foods and dairy products with reduced or no calorie content. And, these low fat food products are gradually becoming a popular choice saturating the market shelves.[3,4] However, many if not most of these low or fat free products fail to thrive as they cannot mimic the sensorial properties of their full fat counterparts.[3,5] It has been demonstrated repeatedly that in case of dairy products, the consumers' liking is positively correlated to creaminess.[6,7]

In past decades, rheology has been used as a "gold standard" instrumental technique to map or predict the perceived texture and mouth feel of dairy products. In other words, most previous studies attempted to mimic the bulk rheological properties of full fat counterparts with an objective of simulating the creaminess perception of the fat free versions.[8–11] However, limited research has been undertaken with employment of appropriate oral conditions (physicochemical and thermal conditions) during these rheological measurements. Hence, bulk and shear rheological studies with addition of artificial saliva at 37 ○C is needed to provide further insights on sensory perception.

It is worth recognizing that creaminess is a complex multimodal sensorial attribute that cannot be simply predicted by rheological parameters. Kokini and co-workers[12,13] pioneered the concept of oral tribology by introducing the regression ana-

[a]*Food Colloids and Processing Group, School of Food Science and Nutrition, University of Leeds, LS2 9JT, UK. E-mail: A.Sarkar@leeds.ac.uk*
[b]*School of Mechanical Engineering, University of Leeds, LS2 9JT, UK*

lysis of creaminess, which not only included rheological parameter, such as thickness but also thin-film tribological parameter as shown in eqn (1) and (2):

$$\text{creaminess} \propto (\text{thickness})^{0.54} \times \text{smoothness}^{0.84} \quad (1)$$

$$\text{smoothness} = 1/(\mu F_{\text{Tongue}}) \quad (2)$$

where, μ is the coefficient of friction between the tongue and the oral palate and F is normal force of the tongue on the food.

Krzeminski and coworkers found positive correlations between destructive rheological parameters and oral viscosity in yoghurts, and pointed out that their predictive model for creaminess suffered from lack of surface-related measurements taking place at a later stage of oral processing.[14] Tribology measurements have been a relatively recent undertaking in oral processing and sensory prediction work in model colloidal systems and dairy products.[11,15–18] Among the recent studies, Selway and Stokes[15] successfully demonstrated that lubrication measurements (μ = 0.06 for high/medium fat, μ = 0.35 for low fat yoghurts) using soft silicone elastomeric tribo-pairs can be used to differentiate rheologically similar yoghurts. Stribeck curves clearly discriminated the cream cheese of different levels of fat contents (0.5%, 5.5%, 11.6%), although their η_{50} apparent viscosities showed no significant difference.[19] However, it is worth pointing out that the rheological measurements performed in these studies did not use simulated oral conditions and no sensory evaluation was carried out on the same commercial low/medium/high fat yoghurts. Hence, the question still remains whether consumers would be able to discriminate those rheologically similar but tribologically different dairy products of different fat contents or not.

Interestingly, most researches dealing with rheology-sensory or tribology-sensory relationship have employed trained panellists to investigate sensory perceptions of dairy products using quantitative descriptive analysis (QDA™).[9,10,20] However, for gaining insights from a more real-life setting, a discrimination test involving a representative general population of untrained males and females is more appropriate. Such tests will help to better understand the consumers' perceived differences (if any) between the full fat and fat free dairy products and whether rheology or tribology under simulated oral conditions can predict those discrimination.

Hence, in the present work, we have combined for the first time, viscoelasticity and flow behaviour, tribology and sensory discrimination test using untrained panellists to differentiate between fat free/low fat and full fat versions of liquid (milk) and semi-solid (yoghurt, cream cheese) colloidal systems. We have simulated the oral environments during rheology and tribology measurements using artificial saliva containing pig gastric mucin at 37 ○C. The attributes used by the consumers to differentiate between fat free/low fat and full fat versions of product classes were also investigated. The null hypothesis for this study was that bulk rheological properties cannot predict the sensory perception, even in the presence of artificial saliva at 37 °C.

2. Experimental

2.1 Materials

2.1.1 Dairy products. Commercial dairy products were purchased from a local supermarket. Morrison's British milk (whole milk 3.6 wt% fat and skim milk 0.1 wt% fat), Yeo Valley Natural yoghurt (full fat yoghurt, 4.2 wt% fat and fat-free yoghurt, 0 wt% fat) and Philadelphia soft cream cheese (full fat cream cheese, 21.5 wt% fat and low fat cream cheese, 2.5 wt% fat) were used. The products were stored at 4 ± 1 °C in their packaging until their characterization.

2.1.2 Artificial saliva. The reagents used for making the artificial saliva were purchased from BDH Chemicals (BDH Ltd, Poole, England) unless otherwise specified. Porcine gastric mucin Type II (Sigma Chemical Co., St Louis, MO, USA) contained 1% bound sialic acids. Milli-Q water (water purified by treatment with a Milli-Q apparatus; Millipore Corp., Bedford, MA, USA) was used as the solvent for saliva preparation.

2.2 Methods

2.2.1 Preparation of artificial saliva. Artificial saliva containing 3 g L^{-1} mucin was prepared according to the composition used in the previous literatures[21,22] by mimicking the ionic composition, rheology and pH of saliva. Artificial saliva and the samples were mixed gently in 1 : 1 w/w ratio based on the oral processing protocol of the standardised static *in vitro* digestion method.[23] Briefly, unstimulated salivary flow rate is 0.3 mL min^{-1} but stimulated flow rate is, at maximum, 7 mL min^{-1}.[24] Nearly, 80–90% of the average daily salivary production is stimulated saliva and thus, based on stimulated salivary flow rate, the mixing ratio of 1 : 1 w/w was selected. It is worth noting that this mixing ratio might vary depending upon the consumed food texture, oral residence time and also might differ during course of oral processing from intake to swallowing beside other physiological and inter-personal factors. However, this dynamic profile of saliva incorporation in the food consumed is not taken into account within the scope of this study.

2.2.2 Small deformation rheology. The rheological properties of the samples were analysed using dynamic oscillatory measurement in a Kinexus rheometer (Malvern, UK). The rheometer was equipped with a 30 mm parallel plates and a gap of 1 mm was selected for all samples. Samples were placed on to the plates using a spatula, and a fresh sample was loaded for each measurement. A temperature cover was used to maintain the samples at the specified temperature, to avoid evaporation. A strain sweep test from 0.01–100% was carried out to determine the linear viscoelastic region at constant angular frequency of 1 Hz. Frequency sweeps were conducted from 0.1–10 Hz at constant strain of 0.1%. To study the differences in viscoelasticity between samples, G' (storage modulus), G'' (loss modulus) and tan δ (G''/G') at 1 Hz, where δ is the phase angle were determined during the measurements were compared. Frequency of 1 Hz was selected because it was considered a reasonable compromise between measuring a very high frequency at which entanglements could contribute to solid-like response and measuring at extremely low frequencies where loss of precision and reliability could occur.[25] Flow curves were obtained for the milk, yoghurt and cheese samples as such and in presence of saliva as a function of shear rate ranging from 0.01–100 s^{-1}. Data from the flow curves were fitted to the Ostwald de Waele fit ($\sigma = K\dot{\gamma}^n$), where K (Pa s^n) is the consistency index and n is the flow index. Tests were carried out on all dairy products with and without the addition of artificial saliva. A temperature of 25 °C was used for all tests as samples were served in the sensory test at this temperature condition. Use of 37 °C was employed for tests with the addition of saliva to simulate oral conditions.

2.2.3 Particle size measurements. The particle size distribution of the dairy products was measured by static light scattering (Malvern MasterSizer 3000, Malvern Instruments Ltd, Worcestershire, UK). The relative refractive index (N) of the dairy products was 1.09, *i.e.* the ratio of the refractive index of milk fat (1.46) to that of the dispersion medium (1.33). The absorbance value of the emulsion particles was 0.001. A regular spherical shape of the fat particles was assumed. The Sauter-average diameter, d_{32} (= $\sum n_i d_i^3/\sum n_i d_i^2$), where n_i is the number of particles with diameter d_i) of the emulsion droplets was measured. All the measurements were performed in triplicate.

2.2.4 Tribology. The tribological properties of all the commercial dairy products was assessed using a Mini Traction Machine (MTM, PCS instruments, UK) to facilitate a mixed rolling and sliding contact. Hydrophobic polydimethylsiloxane (PDMS, Sylgard 184, Dow Corning, USA) tribo-couples were used consisting of a flat plate and Ø19 mm ball (Fig. 1). The surface roughness of the balls and plates was measured using white light interferometry and determined to be R_a = 100 nm. Prior to each test, surfaces were cleaned with acetone and rinsed with ultrapure water. For each test, a new plate was used each time whilst balls were rotated at 180 degrees on the horizontal plane ensuring the same surface was not tested more than once. A normal load of 2 N was used in all tests achieving a maximum Hertzian contact pressure (P_{max}) of ~100 kPa. In each test, sliding speeds were varied from 1000 to 1 mm s^{-1} at a sliding-to-rolling ratio of 50%. Characteristic traction coefficient *vs.* sliding speed curves (*i.e.* Stribeck) for all samples were collected. The entrainment speed of the rolling sliding contact was calculated using eqn (3) (Fig. 1).

$$\bar{U} = \frac{1}{2}(U_1 + U_2) \quad (3)$$

where, $\bar{U}$ is the entrainment speed, U_1 and U_2 are the velocities of the two contacting surfaces (*i.e.* ball and plate). All tests were carried out at 37 °C ± 1 and for three repetitions.

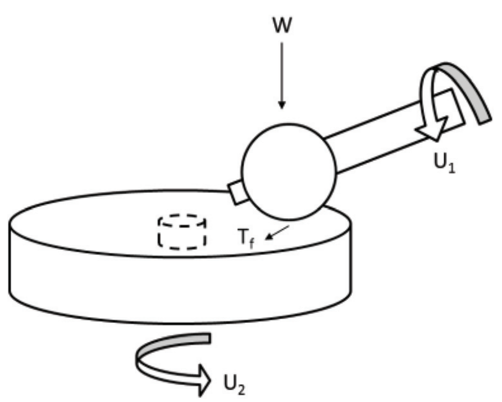

Fig. 1 Illustration of traction tribometer used in this study. W is the normal load, T_F is the traction force exerted by the disk and ball, U_1 and U_2 are the ball and disk speed, respectively.

2.2.5 Sensory test. Milk, yoghurt and soft cream cheese samples were evaluated by 63 untrained consumers (31 males, 32 females, mean age: 24 years) at the Food Technology Laboratory at The University of Leeds, Leeds, UK. The study has been reviewed and approved by Faculty Ethics committee at University of Leeds [ethics reference (MEEC 15-007)].

The participants were not trained but they received instructions regarding the evaluation procedure in both written and verbal format prior to sample evaluation. Consumers (or also called "untrained panellists") gave written informed consent before the start of the study. Consumers sat in partitioned sensory booths, the lighting and temperature of all booths were standardised. Each consumer attended one 30–45 minute session, they had a break of 2–3 minutes between each set of samples (milk, yoghurt, cheese) and they were instructed to take additional breaks if they needed. The presentation order was randomized across consumers. Each sample (10 g) was presented in small clear plastic and odourless cup coded with randomized three digit numbers placed on a white plastic tray. Consumers were provided with white plastic spoons, neutral tasting wafers, and a cup of mineral water, for mouth rising between tastings. All sessions were carried out in (11:00–13:00) in separate booths. The questionnaire given to the consumers had three different parts:

I. Consumption frequency of the products, and type of products they consumed (skim, semi-skimmed or full fat)

II. Triangle test

Untrained panellists were presented with three samples simultaneously. In each set, two samples had the same fat content and one sample had different level of fat – half of the consumers were provided with two full fat and one low fat dairy product, and the other half were given two low fat and one full fat product.

The following instructions were placed on the paper ballot: "Taste the samples from left to right. Two of the samples are identical. Determine which one is the odd sample?"

Then, panellists were asked to give reasons on how they have discriminated the samples, using as many words or phrases as they needed to explain the differences between samples.

III. Intensity score with elicited vocabulary

Panellists used their discriminative vocabulary generated in the triangle test to score the perceived intensity of their discriminative attributes. They chose adjectives to describe appearance, mouth feel, after feel and taste and rated the intensity of each sample based on these attributes on a line scale. The ratings were converted to a number from 0 (left) to 10 (right) (0 = not at all, and 10 = very).

2.2.6 Statistical analysis. Means and standard deviations of rheology and tribology experimental values were calculated. Rheological parameters with different fat content and presence of saliva were studied by a descriptive one-way ANOVA, the least significant differences were calculated by Tukey test and the significance at $p < 0.05$ was determined. For sensory analysis, all results for the discrimination test were recorded. Only data on intensity ratings was evaluated for consumers who had correctly identified the odd sample. The most commonly used adjectives to describe appearance, mouth feel, after feel and taste were recorded and a paired comparison *t*-test was carried out to determine if there were significant differences at $p < 0.05$ between full fat and low fat variants of each product classes. Tests were done using SPSS (IBM SPSS Statistics for Windows, Version 22.0. Armonk, NY: IBM Corp).

3. Results and discussion

3.1 Particle size distribution

It is well known that particle size might influence the sensory perception. Hence, the particle size distribution of milk, yoghurt and cheese samples with varying fat percentage is shown in Fig. 2. Skim milk (0.1 wt% fat) showed monomodal distribution with peak at around 0.15 μm while the whole milk was bimodal with peaks in 0.15 μm as well as in 0.8 μm (Fig. 2A), which is consistent with previous literature value.[19] The first peak in both the skim and whole milk corresponds to free casein micelles[26,27] and the second one in case of the whole milk represents the fat globules,[28] which is consequently absent in the skim milk, later resulting in difference in d_{32} values. This suggests that fat replacer particles of similar particle size to fat droplets were not added in the skim milk. In case of yoghurt and cheese (Fig. 2B and C), both no/low and high fat versions contained similar range of particle size with single peak containing particles in the range of 1–100 μm, which suggests that the fat mimetics used in the low/no fat systems might have similar range of particle size as that of the milk fat globules. It is worth noting that lubrication properties of fat replacer particles can be explained by "ball-bearing effects" of spherical shaped and small sized particles.[29] Hence, low fat and full fat versions with similar particle size might be hypothesized to have similar lubrication and sensory aspects.

3.2 Bulk rheology

3.2.1 Milk. Flow curves were obtained for whole and skim milk at 25 °C and after the addition of saliva at 37 °C. Fig. 3 show that both whole and skim milk samples had low viscosities (~0.1 Pa s)[19] and had overlapping trend. As shear rate increased, the viscosity of both the milks decreased, showing shear thinning behaviour with almost identical apparent viscosity values irrespective of their fat content, which is in agree-

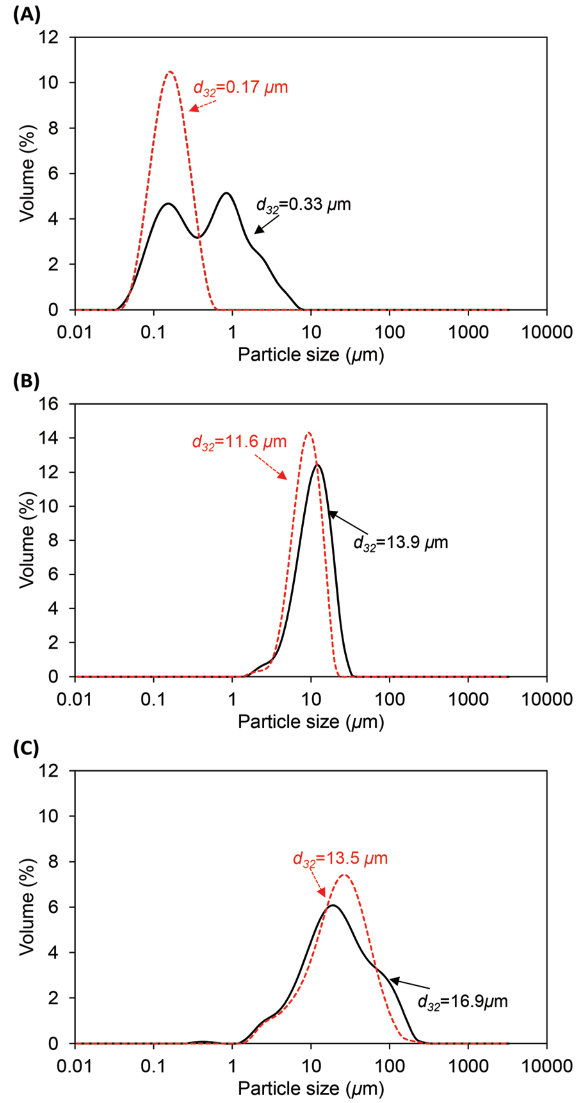

Fig. 2 Particle size distribution of full fat (solid) and low/no fat (dashed line) versions of milk (A), yoghurt (B) and cheese (C), respectively.

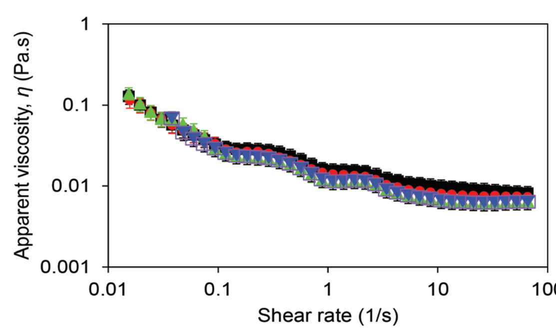

Fig. 3 Flow curves of whole (3.6 wt% fat, ■) and skim (0.1 wt% fat, ●) milks at different shear rates in absence or presence (whole ▲, skim ▼) of artificial saliva, respectively. Error bars represent standard deviations.

ment with previous report.[19] The addition of artificial saliva appeared to slightly reduce the viscosity of both the milks, though not significant ($p > 0.05$), and, the overlapping shear thinning behaviour of both whole and skim milk became even more prominent. Overall, it can be inferred, that there was no significant difference ($p < 0.05$) in flow behaviour and consistency index of whole and skim milk even on addition of saliva (Table 1).

3.2.2 **Yoghurt.** For yoghurt, the apparent viscosity values were in a considerably higher range (up to 500 Pa s as compared to less than 1 Pa s for milks) (Fig. 4). As expected, yoghurts showed a very typical shear thinning (pseudoplastic) flow behaviour as shear rate increased.[30]

No significant difference in viscosity at 50 s^{-1} (relevant to oral shear) was observed between the two yoghurt samples, despite variations in fat and protein content highlighting that fat might not have any significant role on flow behaviour in set-yoghurt.[15]

The other obvious hypothesis might be that the no-fat yoghurt has been formulated in such a way that it exactly matches the apparent viscosities of the full fat counterpart. Based on different functionalities of fat in texture and mouth feel, three kinds of fat replacers are known: thickening agents to control rheological properties, bulking agents to increase adsorption to the tongue, and microparticulated ingredients to enhance lubrication properties.[31] Considering that ingredient list does not highlight any particular ingredient in the no-fat yoghurt, one might suggest that processing of the dairy ingredients might be contributing to similar viscosities as well as matching the size of fat droplets as shown in previous section. As it might be expected, on addition of artificial saliva, the apparent viscosities of the yoghurt/saliva mix had an intermediate value between yoghurt and saliva viscosity, which might be attributed to the dilution effect as well as shear thinning behaviour of mucin.[32,33] However, there was no significant difference between the viscosities of full fat and fat-free yoghurt (Table 1) under this simulated oral condition. Viscoelastic materials, such as yoghurt can be adequately described by two parameters, the storage modulus (G') which is a measure of its elastic nature, and the loss modulus (G'') which is a measure of its viscous nature.[34] Fig. 5 shows the mechanical spectra of the full and no fat yoghurts in absence and presence of saliva, respectively.

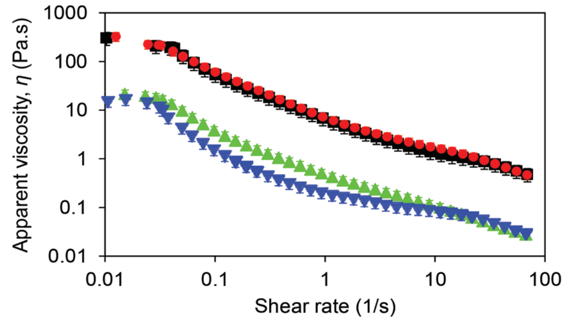

Fig. 4 Flow curves of full fat (4.2 wt% fat, ■) and fat-free (0 wt% fat, ●) yoghurt at different shear rates in absence or presence (full fat ▲, fat-free ▼) of artificial saliva, respectively. Error bars represent standard deviations.

Both full fat and no fat yoghurt samples showed typical characteristics of weak viscoelastic colloid gel (Fig. 5A), with dominance of G' over G'' and no significant difference in G'. The G' and G'' in all the yoghurt samples were independent of frequency across the range of frequencies studied. As it can be observed in Table 1, no significant differences were found in the $\tan\delta$ for yoghurts with different fat concentrations with values similar to previously reported values.[15] The linear viscoelastic region (LVER) was slightly larger for full fat (γ = 0.01–3%) than the fat-free (γ = 0.01–1%) yoghurt samples (stain sweep data not shown). The G' was significantly below G'' at strain ~5% for fat-free yoghurt, whereas for full fat, the crossover was at a relatively higher strain (~20%). This might

Table 1 Rheological parameters of the milk, yoghurt and soft cream cheese samples with different fat contents. Consistency index (K), flow index (n), $\tan\delta$, storage modulus (G') and loss modulus (G'') values are given as average values of three measurements ± SD (α = 0.05). Means (in the same column) with the same letter do not differ significantly ($p < 0.05$) according to Tukey test

	Ostwald de Waele fit ($\sigma = K\dot{\gamma}^n$)		Viscoelastic parameters measured at 1 Hz		
Dairy products	K (Pa s^n)	n	G'	G''	$\tan\delta$
Whole milk or Full fat milk (3.6 wt% fat)	0.027 ± 0.001^a	-0.530 ± 0.013^a	—	—	—
Skim milk or low fat milk (0.1 wt% fat)	0.024 ± 0.002^a	-0.536 ± 0.066^a	—	—	—
Full fat milk + artificial saliva (37 °C)	0.021 ± 0.001^a	-0.533 ± 0.008^a	—	—	—
Low fat milk + artificial saliva (37 °C)	0.025 ± 0.004^a	-0.661 ± 0.115^a	—	—	—
Full fat yoghurt (4.2 wt% fat)	8.385 ± 0.854^b	-0.750 ± 0.063^a	294.35 ± 58.05^c	72.3 ± 13.78^c	0.255 ± 0.097^a
Fat free yoghurt (0 wt% fat)	9.455 ± 2.343^b	-0.769 ± 0.049^a	240.25 ± 78.56^b	65.30 ± 22.03^b	0.271 ± 0.003^a
Full fat yoghurt + artificial saliva (37 °C)	0.634 ± 0.246^a	-0.796 ± 0.079^a	1.83 ± 2.28^a	0.62 ± 0.65^a	0.534 ± 0.311^a
Fat free yoghurt + artificial saliva (37 °C)	0.333 ± 0.121^a	-0.720 ± 0.012^a	0.83 ± 0.64^a	0.47 ± 0.20^a	0.681 ± 0.284^a
Full fat cheese (21.5 wt% fat) (37 °C)	90.84 ± 8.468^c	-0.861 ± 0.002^a	4770.52 ± 746.20^c	1087.9 ± 201.12^d	0.224 ± 0.001^{ab}
Low fat cheese (2.5 wt% fat) (37 °C)	250.56 ± 13.661^b	-0.885 ± 0.000^a	3739.45 ± 857.24^b	996.48 ± 248.57^c	0.261 ± 0.005^{ab}
Full fat cheese + artificial saliva (37 °C)	41.82 ± 5.215^a	-0.763 ± 0.119^a	69.07 ± 28.21^a	14.38 ± 5.74^b	0.188 ± 0.002^a
Low fat cheese + artificial saliva (37 °C)	66.28 ± 0.001^{ab}	-0.755 ± 0.000^a	18.90 ± 9.70^a	8.61 ± 4.12^a	0.406 ± 0.090^b

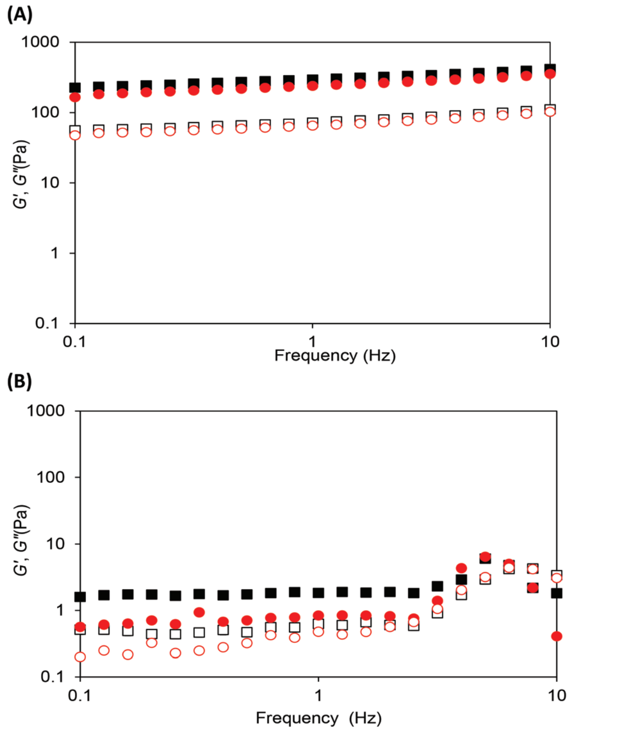

Fig. 5 Storage modulus (G', closed symbols) and loss modulus (G'', open symbols) of full fat (■) and fat-free yoghurts (●) in absence (A) or presence or artificial saliva (B), as a function of frequency at constant strain of 0.1% respectively.

be attributed to the absence of fat globules acting as structure promoters or "active fillers" of the protein network in case of the fat-free yoghurt, resulting in a lower elastic behaviour (lower G' value).[35,36] Addition of saliva to the yoghurt significantly reduced G' and G'' ($p < 0.05$) resulting in weakening of the gel structure (Fig. 5B).

On addition of saliva, the difference between G' and G'' values for in full fat and fat-free yoghurt was abridged, particularly at high frequencies (>4 Hz). In presence of saliva, both the full and fat-free yoghurts became more liquid like ($\tan\delta > 0.5$). Rheological parameters, such as yield stress, viscosity and elastic modulus define the bulk properties of yoghurt at extremely low shear rates, up to the point of flow. Many previous studies have correlated these instrumental parameters to several different sensory attributes.[35,37] So, intuitively based on iso-rheological properties it might be hypothesized that sensorially there would be no significant difference between the full fat and fat-free versions of yoghurt when tested with untrained consumers.

3.2.3 Cheese. Fig. 6 shows the dynamic viscosity curves of low fat (2.5 wt%) and full fat (21.5 wt%) cheese, respectively, as a function of shear stress. The yielding process of cheese occurred over a wide range of shear stress values, reflecting the behaviour of highly pseudoplastic fluids with finite zero-shear viscosities.

Unlike milk and yoghurts, the apparent viscosities of the full fat cheese were significantly higher as compared to low fat

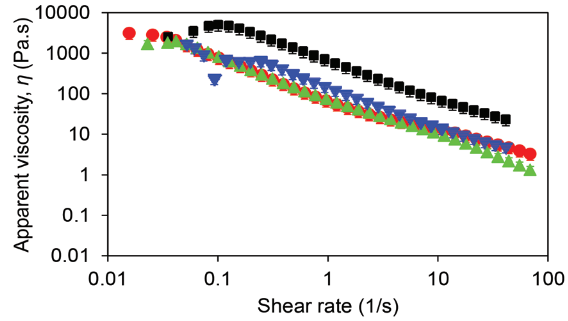

Fig. 6 Flow curves of full fat (21.5 wt% fat, ■) and low fat (2.5 wt% fat, ●) cheese at different shear rates in absence or presence (full fat ▲, low fat ▼) of artificial saliva, respectively. Error bars represent standard deviations.

cheese (Fig. 6, Table 1). However, on addition of artificial saliva at 37 ○C, there was no significant difference in the flow curves of full fat and low fat cheese ($p > 0.05$), which might be attributed to dilution, as well as interactions with highly elastic saliva containing shear-dependent mucin molecules. Full fat soft cream cheese had a slightly but significantly higher G' and G'' than its low fat counterpart (Table 1, $p < 0.05$), which gives an indication of higher number and strengths of fat droplet ("active filler")-protein matrix interaction in the former. Both soft cheese samples had G' consistently higher than G'' suggesting a dominance of solid behaviour (Fig. 7A).

The frequency tests show that G' of low fat and high fat versions of cream cheese without the addition of saliva were independent of frequency. In presence of artificial saliva, (see Table 1, $\tan\delta$), the sample with low fat content presented the highest liquid-like behaviour. This means that in the case of the low fat cheese, its oral processing (in presence of saliva and a 37 °C) may be considerably different than the full fat version. The LVER of the strain sweep curve of the low fat cream cheese reached 1% strain; after which the G' and G'' started to fall (strain sweep data not shown). However, for full fat cream cheese, the LVER reached 10% strain before the catastrophic fall, suggesting the full fat cheese had taken a moderately higher strain to break. Although, the addition of saliva did significantly reduce the magnitude of G' and G'' for both the samples, the trend of the curves remained similar with more significant difference in G' between full fat and low fat cream cheese even at higher frequencies ($p < 0.05$), (Fig. 7B). Besides mucin, the contributory factor in the reduction in G' and G'' (Fig. 7A and B) in presence of saliva may be the difference in temperature employed in the rheology tests (without saliva at 25 °C, *versus* with saliva at 37 °C). The oral heating used might have caused melting of the fat and thus a decrease in the G'.[38] In summary, despite variation in fat and protein contents, samples within each product series (*i.e.*, milk and yoghurts) exhibited similar bulk rheological behaviour, with the exception of cream cheese. The cream cheese tested showed slight but statistically significant ($p < 0.05$) difference

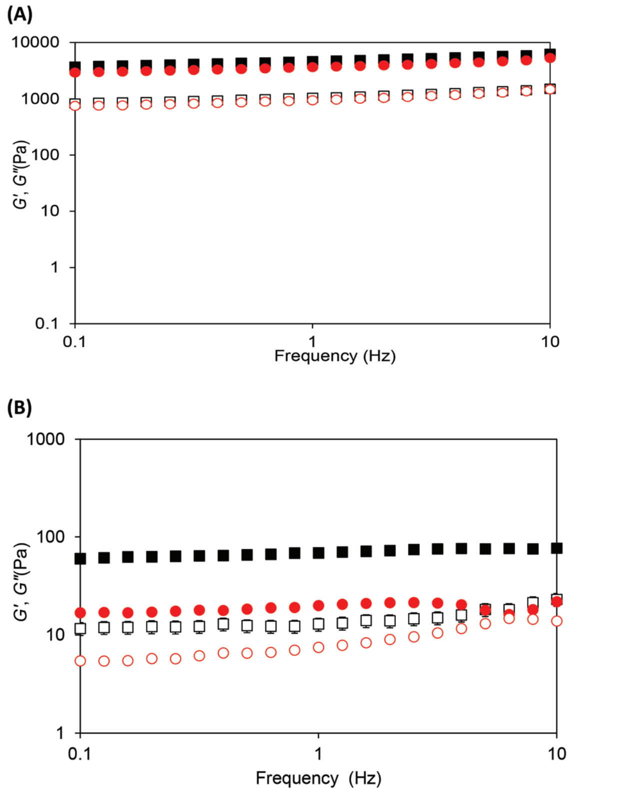

Fig. 7 Storage modulus (G', closed symbols) and loss modulus (G'', open symbols) of full fat (■) and low fat cream cheese (●) in absence (A) or presence or artificial saliva (B), as a function of frequency at constant strain of 0.1% respectively.

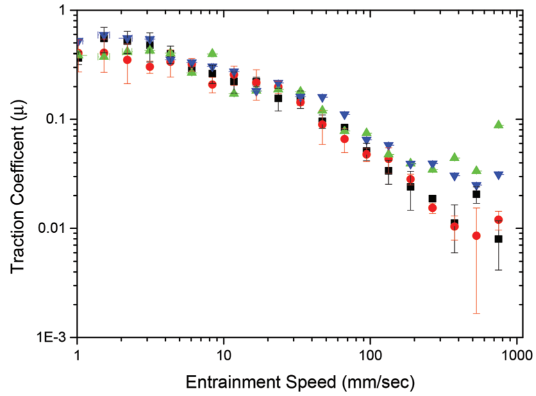

Fig. 8 Traction coefficient dependence of milk samples at variables speeds for whole milk (■), skim milk (●), whole milk + saliva (▲) and skim milk + saliva (▼).

in elastic modulus and yielding properties between full fat and low fat variants in both presence and absence of saliva. This small distinction between the low and full fat cream cheese samples may translate to distinct mouthfeel sensations.

3.3 Tribology

3.3.1 Milk. Stribeck analysis allows for the speed dependant lubricating film formation to be determined for a certain set of contacts and lubricants. Fig. 8 shows the Stribeck analysis for whole and skim milks with and without the addition of artificial saliva. A speed dependent traction coefficient could be observed in these tests. The PDMS contacts transitioned from a boundary (*i.e.* surfaces in contact) to mixed lubrication regime (*i.e.* partial contact with the onset of EHL (elastohydrodynamic lubrication)) been observable with increasing entrainment speed. The addition of saliva was seen to have no significant effect on the boundary and mixed regime ($p > 0.05$). At higher entrainment velocities, deviation in the curves was observed for both samples containing saliva, with significantly higher traction coefficients been observed. This is in contrast to the data obtained by previous studies, which observed a clear discrimination between samples of different fat contents (even between 0.1 wt% and 2.0 wt% fat content, lower than the difference levels in fat tested in the current study) at all investigated entrainment speeds.[19] Chojnicka-Paszun and coworkers[39] identified that the traction coefficient measured for idealised milks was a function of the tribo-couple used (neoprene o-ring on silicone/neoprene/Teflon) as well as the fat content. Hence, it must be noted that friction responses are highly system dependant (both surfaces and lubricant). The difference in contact surfaces of PDMS used in our study *versus* hydrophobic rough surface using 3 M Transpore Surgical Tape 1527-2 [19] or Teflon/Noprene surfaces[39] can also result in different Stribeck curves with the same lubricants.

As the aim of this research was to relate rheology and tribology of commercial dairy colloids to sensory perception, no effort to regulate particle size or protein content was made. It can be expected that if a fat droplet mediated boundary lubrication type mechanism is present, surface roughness, contact area and particle size and concentration will have a significant role on modifying the lubrication processes. Tribology analysis on commercially available milks was unable to differentiate between milk samples, which suggests that the mechanisms of lubrication are more complex and multifactorial. More research into these tribological and colloidal variables and their synergies is needed and tongue surfaces needs to be mimicked accurately to understand oral lubrication in greater depth.

3.3.2 Yoghurt. Fig. 9 shows the traction coefficient dependence with entrainment speed of yoghurt samples with and without saliva. Significant differences in traction coefficients were observed between fat-free and full fat products ($p < 0.05$). Lower traction coefficients were observed for the full fat yoghurts ($\mu \sim 0.05$) when compared to fat-free ($\mu \sim 0.4$–0.6) at lower entrainment velocities (<10 mm s^{-1}), correlating with the work of Selway and Stokes.[15] A decrease in friction with entrainment speed was observed in both samples. However, for fat free yoghurts no transition to an EHL regime could be observed. At higher entrainment velocities this can be explained by the significant reduction in apparent viscosity (Fig. 4), prolonging the transition into the EHL regime due to the additional fluid pressurisation required to separate the

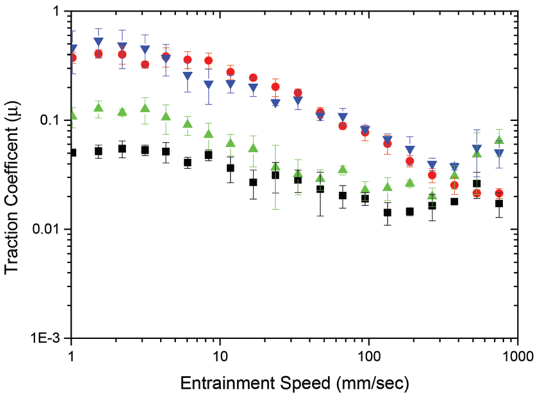

Fig. 9 Traction coefficient dependence of yoghurt samples at variables speeds for full fat (■), fat free (●), full fat + saliva (▲) and fat free + saliva (▼) yogurts.

contacting surfaces. The addition of saliva was not seen to have a significant effect on the traction coefficients for fat free yoghurts. However, it increased the traction coefficient significantly in boundary and EHL regimes for the full fat yoghurt. A prolong boundary regime for the full fat yoghurts, when compared to fat free yoghurt, was observed. This suggests a boundary lubrication mediated mechanisms may be present.[19] Fat droplets are thought to coalesce within the tribological contact surfaces reducing the traction coefficient until a sufficiently high shear is established to disrupt any boundary layers.

3.3.3 **Soft cream cheese.** Similar observations were made for the soft cream cheese (Fig. 10). Clear and distinctly identifiable boundary, mixed and EHL regimes were observed for the high fat containing cheeses with and without saliva. Low fat cream cheese markedly increased the traction coefficient ($p < 0.05$) in both the boundary and mixed lubrication regimes. Comparing to slight differences in rheology results (Fig. 7A and B), the Stribeck curves (Fig. 10) of full fat and low fat cheese showed almost two-orders of magnitude difference at low entrainment speeds. On average, the addition of saliva was seen to increase friction coefficients although not significantly. The same mechanism for milks has been applied to such semi-solids in which a fat droplet mediated boundary lubrication type mechanisms exists within the tribological contact. It is hypothesised that fat droplet may coalesce within the contact, reducing friction through a boundary layer type lubrication. To date no evidence has been presented confirming if this is through a physical (*i.e.* particles within a soft contact), chemical (bonding of fats to the surface) or a tribo-chemically induced process (tribology-induced chemical reactions).

Fig. 11 shows the traction coefficient as a function of entrainment velocity for artificial saliva. A decrease in traction coefficient with increasing speed could be observed although no identifiable transition to mixed or EHL regimes could be observed. When compared to the work of Bongaerts *et al.*,[40] the artificial saliva was seen to impart superior lubricating properties within the PDMS contacts when compared to PDMS contacts in water. This could be in part to a slight increase in the apparent viscosity but likely dominated by the ability for salivary proteins *i.e.* mucins to act as an effective boundary lubricant.[41]

In summary, tribology evaluation of the semi-solids has been able to clearly and significantly discriminate semi-solid emulsion gels *i.e.* yoghurts and cream cheese with different fat contents, which was not observable in rheological evaluation in yoghurt and was not very clear in case of cheese samples. As discussed before, the particle size measurement could not identify significant differences between the low fat/fat-free and full fat versions in case of yoghurt and cheese. Although the fat-replacer added in the low or no fat versions might have similar particle size to that of fat droplets, differences in surface roughness or irregularities contributed by such ingredients might have influenced the lubrication properties.[42] Particularly, in case of low fat cream cheese, the presence of hydrocolloids, such as carob gum and carrageenan might also have resulted in higher traction coefficients, which needs to be further studied using model systems. Furthermore, coalescence of fat droplets might not have occurred in the low fat or fat-free systems, which might be responsible for difference in

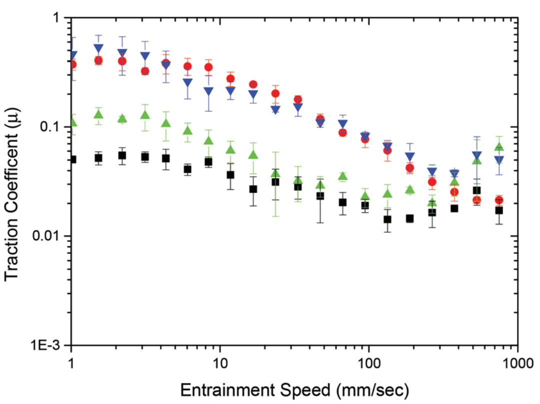

Fig. 10 Traction coefficient dependence of cream cheese samples at variables speeds for full fat (■), low fat (●), full fat + saliva (▲) and low fat + saliva (▼) cream cheese.

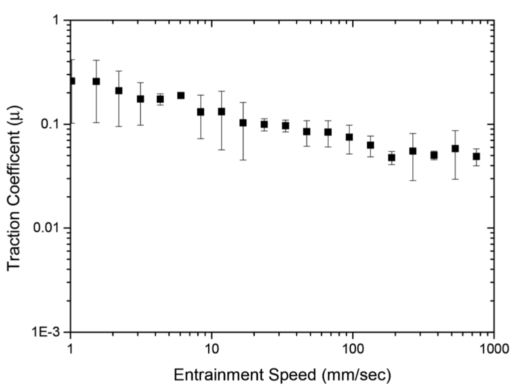

Fig. 11 Traction coefficient dependence of artificial saliva at variables speeds.

traction coefficients. Significant difference in boundary lubrication regimes was observed with traction coefficients converging at different fat levels. This further supports the hypothesis that fat-mediated boundary lubrication might play a role in mouth feel by reducing the traction coefficient and prolonging the point in which the lubrication regimes transitions from a boundary to mixed regime. The fat content was also seen to extend mixed lubrication regimes. This suggests that the fat droplets might play a role in pressurisation of bulk fluid within the contact that is required to separate the surfaces at higher entrainment velocities. Further work to identify these mechanisms is currently underway.

3.4 Sensory analysis

Paper ballots with the frequency of consumption, discrimination test and rating scales were used to collect sensory data. For triangle test with 60 responses, the minimum number of correct responses required for significance at $p < 0.001$ is 33.[43,44] Table 2 shows that number of untrained panellists who were able to discriminate between full fat and fat free/low fat dairy products were statistically significant.

The results of the ratings were recorded and the most commonly used adjectives to describe the four sensory attributes across those consumers who were able to discriminate between fat contents can be seen in Table 3. Consumers chose a greater variety of adjectives for mouth feel and after feel, hence, the next most commonly used adjective has been included for these attributes in Table 3.

3.4.1 Milk. As it can be observed in Table 3, the most commonly used adjective to describe the difference in milk appearance was 'white'. 'Creamy' and 'watery' were the most commonly used adjectives to describe the difference in mouthfeel. Whole milk had a significantly higher ($p = 0.001$) intensity rating for 'white' than skim milk at significance (Table 3). Whole milk was scored as more creamy and less watery ($p = 0.0011$, $p = 0.002$) than skim milk. After feel attributes were also significantly discriminated for milk. The most frequently used after feel adjectives were 'watery' and 'creamy'. Consumers were able to distinguish between the whole and skim milk samples significantly (Tables 2 and 3). Skim milk had a significantly higher mean intensity rating for 'watery' ($p = 0.004$), and significantly lower ratings for 'creamy' than whole milk ($p = 0.001$). However, ratings for 'sweet' taste were not significantly different ($p = 0.916$) (Table 3).

Table 2 Number of correct responses for sensory analysis using a discrimination test for milk and yoghurt

Product	Number of correct responses	Total number of responses
Milk	39*	62
Yoghurt	39*	63
Soft cream cheese	37*	63

*Significance at 0.1%.[43,44]

3.4.2 Yoghurt. As seen in Table 3, mean intensity ratings for 'white' appearance of yoghurt were not significantly different between full fat and fat-free yoghurt. Likewise, the mean intensity ratings for the two most common mouth feel adjectives ('creamy' and 'thick') and after feel adjective 'creamy' were not significantly different between the full fat and fat free yoghurt ($p > 0.05$). On the other hand, untrained panellists rated 'slimy' after feel to be significantly lower for full fat yoghurt compared to the fat free counterpart ($p = 0.029$). 'Sour' was used by 34 untrained panellists to describe the taste of yoghurt; however, the mean intensity ratings were not significantly different between fat contents (Table 3). From these results, it can be seen that whilst the majority of untrained panellists were able to discriminate between fat free and full fat yoghurt (Table 2), which were iso-rheological but tribologically significant different, the sensory significant difference was only detected in the 'slimy' after feel. This is in line with previous studies where fat-free or low-fat yoghurts made with inulin[45] or milk proteins[46] showed inferior flavour, consistency and mouth feel attributes, although having similar rheological properties.[45] This suggests that tribology can be a promising method to predict sensory behaviour of emulsion gels.

3.4.3 Soft cream cheese. The most commonly used adjective to describe soft cheese appearance was 'white'. Untrained panellists' intensity ratings for 'white' were not significantly different between full fat and low fat soft cream cheese, ($p > 0.05$) (Table 3). The most prevalent adjectives used for mouth feel were 'creamy' and 'thick'. It is worth noting that ratings for 'creamy' mouth feel were not significantly different between full fat and low fat soft cheese ($p > 0.05$). Untrained panellists' described the after feel of soft cheese as 'fatty'; with full fat scoring a moderately higher average intensity than the low fat counterpart, although not significantly different ($p > 0.05$). However, the average rating of untrained panellists for 'creamy' after feel for full fat soft cheese was significantly higher as compared to that for low fat soft cheese, ($p = 0.019$) as seen in Table 3. 'Sour' was the most commonly used adjective to describe the taste of cream cheese, with low fat soft cheese scoring a higher intensity rating of 'sourness' than full fat but not at a significantly different level ($p > 0.05$).

In summary, the untrained panellists were able to discriminate between full fat and no/low fat versions of the three commercial dairy products, however for milk, they do know the magnitude of the discrimination, and probably the cause. For yoghurt and cheese, they were not able to identify the cause of differentiation, except in afterfeel. This finding suggest that identification of low/fat-free *versus* full fat dairy products is possible by consumers and texture properties were most easy to differentiate in liquid (milks) than in semi-solid (yoghurt, cheese), which is consistent with previous findings.[5] This leads to a key challenge for product developers because untrained panellists are able to discriminate and possibly reject low fat products, but cannot describe the cause of such perception, which remains largely unknown (or insignificant).

Table 3 Sensory evaluation of low/no fat and full fat versions of milk, yoghurt and soft cheese with most popular adjective (italics), number of untrained panellists who correctly discriminated and used that adjective (bold), mean intensity rating, (±) the standard deviation and paired test *p*-value

Product	Appearance	Mouth feel	Second mouth feel	After feel	Second after feel	Taste
Skim milk 	*White* **23**, 5.34 ± 2.54	*Creamy* **19**, 4.66 ± 3.07	*Watery* **7**, 10.53 ± 1.25	*Watery* **9**, 9.03 ± 2.69	*Creamy* **8**, 4.51 ± 1.99	*Sweet* **30**, 6.61 ± 3.22
Whole milk 	9.04 ± 2.05 $p = 0.001$*	7.87 ± 2.81 $p = 0.011$*	4.04 ± 2.90 $p = 0.002$*	3.66 ± 1.88 $p = 0.004$*	9.09 ± 1.72 $p = 0.001$*	6.69 ± 2.80 $p = 0.916$
Fat free yoghurt 	*White* **21**, 7.50 ± 3.31	*Creamy* **13**, 7.21 ± 3.02	*Thick* **12**, 7.71 ± 2.48	*Creamy* **10**, 6.58 ± 2.63	*Slimy* **6**, 8.52 ± 2.08	Sour **34**, 7.89 ± 2.88
Full fat yoghurt 	7.69 ± 2.91 $p = 0.805$	6.92 ± 2.87 $p = 0.780$	6.83 ± 2.60 $p = 0.470$	7.19 ± 2.09 $p = 0.524$	6.07 ± 1.42 $p = 0.029$*	6.70 ± 3.07 $p = 0.169$
Low fat soft cream cheese 	*White* **15**, 8.15 ± 2.12	*Creamy* **18**, 7.10 ± 2.20	*Thick* **8**, 7.16 ± 2.37	*Fatty* **8**, 6.80 ± 2.63	*Creamy* **7**, 6.34 ± 2.62	*Sour* **13**, 7.28 ± 2.77
Full fat soft Cream cheese 	6.43 ± 3.24 $p = 0.100$	8.74 ± 2.61 $p = 0.100$	7.88 ± 2.29 $p = 0.646$	8.38 ± 1.95 $p = 0.274$	9.71 ± 1.66 $p = 0.019$*	5.90 ± 3.36 $p = 0.284$

*Significant at $p < 0.05$.

4. Conclusions

We have presented a combination of rheology, tribology and sensory analysis (with untrained panellists) to identify the differences (if any) between full fat and low/fat-free versions of dairy products in the form of liquid and semi-solid. Majority of untrained panellists were able to statistically discriminate between low fat/fat free versions from the full fat ones in all the dairy product classes, of these a small number were able to describe and rate the differences. We validated the null hypothesis that the rheological tests employed in this study of commercial dairy products were not sufficient to predict sensory evaluations of those products, particularly in case of yoghurt and milk. Although the addition of artificial saliva at 37 °C to the rheology test samples significantly affected the viscoelastic properties, but, no significant differences were established between the bulk rheological properties of the full fat and low fat/fat free versions in these simulated oral conditions, particularly for yoghurt and milk. Typical Stribeck curves obtained clearly discriminated the semi-solid dairy products (yoghurt, cheese) with different fat contents in both presence of absence of saliva. However, tribology could not discriminate the whole and skim milk even in presence of saliva in contrast to literature, although consumers could discriminate and identify the differences in terms of mouthful. It is suggested that a standard protocol for food tribological measurements be adopted to enable proper data comparison among studies. As a conclusion, tribology measurements in presence of artificial saliva appears to be potential technique to more accurately capture the dynamics of oral processing and can be used to unravel insights for texture and mouth feel perception as observed by sensory analysis by consumers, particularly for emulsion gels based systems, such as yoghurts and cheese. The tribological set up will be further investigated to be suitable for predicting sensory differences in thin colloidal liquids, such as milks with suitable contact surfaces.

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