A Flexible Platform for Tribological Measurements on a Rheometer

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ABSTRACT

A newly designed tribological cell for a Rheometer and its application on various samples is described. Tribological measurements on dry, oil and grease lubricated systems have been conducted. Tests on some food products reveal a good correlation between the friction properties and the fat content of the samples.

INTRODUCTION

Compared to oils greases have a number of advantages with respect to the construction and service of lubricated components. However, due to the visco-elastic behavior of greases there are certain constraints to consider. Therefore having an instrument and methods to investigate the visco-elastic and frictional behavior of greases over an extended temperature range is highly desirable. Oscillatory amplitude sweeps are very well suited to investigate the visco-elastic behavior and consistency of lubrication greases. Valuable information on the visco-elastic behavior, i.e. the storage and the loss moduli as well as on the stress values at the flow point, i.e. the yield stress, are obtained[1].

In shear rheology the surface of the fixtures do not have any influence on the rheological data as long as the conditions of laminar flow are met. In some cases the surfaces are treated or roughened in order to prevent slip and to assure a laminar flow field. Therefore rheology uses the test fixtures to apply deformation onto the sample, whereas they are part of the test specimen in tribological testings. However, in tribological tests forces, movements and normal loads need to be applied or measured as in the case of rheology. Modern rotational rheometers are equipped with excellent speed and torque control as well as an accurate normal force detector and a precise control of the temperature in a wide range. All these features can also be used for tribology as well, which led to the idea to design an accessory enabling tribological measurements on a conventional rotational rheometer.

Recently, the lubricating properties of certain liquid and semi-solid foods, as determined with a variety of experimental tribology instruments, was shown to correlate well with mouthfeel attributes[2]. However no standard instruments are currently available. The lubrication behavior of foods has been measured using tribology equipment, e.g. pin-on-disk or a ball-on-disk set up originally designed for industrial applications such as the evaluation of machine wear[2,3]. Ideally, food lubrication would involve the measurements of phenomenon more similar to the actions that occur in the mouth during food consumption. These entail rubbing and squeezing actions of the food item between the tongue and palate, which are not measured by rheology. These rubbing and squeezing motions generate a frictional force in which the food-saliva mixture acts as a lubricant. The oral friction can be described with the help of Stribeck curves, in which the boundary and mixed regimes describe the friction of a food material, which is at least in partial contact with surfaces. As friction is the force that resists motion, it is highly system related and not a material property[4]. Therefore the load, surface and sliding speed at which the experiments are performed are equally important and several investigators have begun studies to find the appropriate conditions. Values obtained from oral measurements show sliding speeds up to 200 mm/s and loading regimes between 0.01 and 90 N[5]. Instruments intended to measure the lubricity of food products must be capable of precise control of test parameters to accurately capture the complex actions that occur in the mouth.

An important aspect in the determination of lubricity is the use of appropriate surfaces. Attempts have been made using tetrafluorethylene (PTFE) and zirconia[6] or steel against silicon rubber[2]. These surfaces, however, are smooth, and do not really mimic the oral tissue of the mouth[3]. More recently, friction coefficients of foods were determined using additional materials that included pig tongues and silicone surfaces and materials with well-defined surface profiles[7,8]. It was found that surface structure critically affects the frictional behavior of the investigated system. Furthermore the importance of the salivary film's interaction with the surface and its influence on the friction measurements are pointed out. The relationship of these surfaces in the prediction of human sensory attributes has still not been fully understood.

A better understanding of the material properties that influence the mouthfeel of dairy products would be of great benefit to the food and beverage industries, especially for low fat and calorie-reduced products. Developing further instrumental techniques to elucidate the physical origins of sensory attributes such as "creaminess" "smoothness" and "thickness" would largely benefit product developers and allow the more consistently delivery of key mouthfeel attributes in their products[9].

TRIBOLOGY DEVICE

For testing tribological properties a new device has been designed, which can be mounted as an accessory onto a standard rotational rheometer[10]. It makes use of the large measurement ranges as well as the motor control mechanism of the rheometer, thus transforming the rheometer into a highly sophisticated tribometer. The setup is based on the ball-on-three-plates-principle (or ball-on-pyramid) consisting of a geometry in which a sphere is held, an inset where three small plates can be placed, and a bottom stage movable in all directions on which the inset can be fixed. Figs.1 and 2 depict the new tribometer setup. The flexibility of the bottom plate is required to get the same normal load acting evenly on all the three contact points of the upper ball. The rotating sphere is adjusted automatically and the forces are evenly distributed on the three friction contacts. An overload of one contact point would result in wrong friction values.

The ball as well as the plates for the inset can be exchanged so that the system can be adapted to desired material combinations. For tribological measurements on food samples elastomers are used as bottom plates. Due to the elasticity of the bottom contact the ball will press into the elastomer and the rotation of the ball will lead to a build up of a lubrication film. This process is called soft elasto hydrodymanic lubrication (Soft-EHL).

The rotational speed applied to the shaft is producing a sliding speed of the ball with respect to the plates at the contact points. The resulting torque can be correlated with the friction force by employing simple geometric calculations. The normal force of the rheometer is transferred into a normal load acting perpendicular to the bottom plates at the contact points. The tribology setup is temperature controlled by Peltier elements from -40°C up to +200°C. A Peltier hood ensures the same temperature at the bottom plates and at the upper ball.

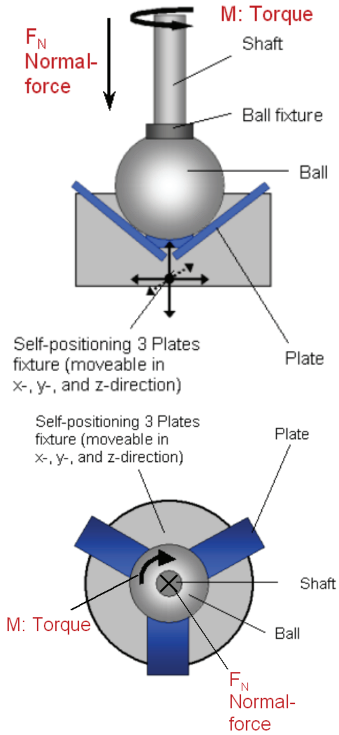

Figure 1. Schematic setup Tribology accessory in side and top view.

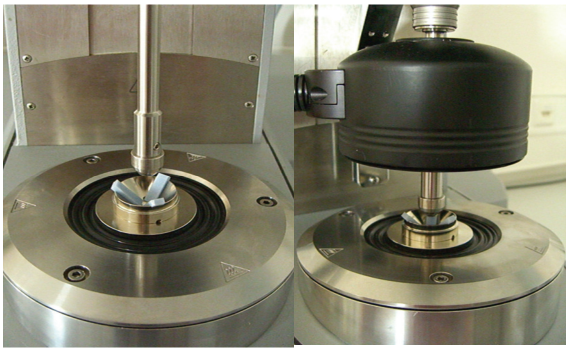

Figure 2. The Tribology accessory without (left) and with (right) additional Peltier hood.

The described device was mounted on a MCR301 rheometer from Anton Paar. Tests with various thermoplastic elastomers revealed that the elastomers suited for tribological testing on food samples having a difference in friction factor of more than 0.6 between an aqueous 10% (w/w) sucrose solution and sunflower oil at sliding speeds in the range of 0.4 and 20 mm/s [11].

MEASUREMENTS ON LUBRICATION GREASES

In Fig. 3 rheological strain sweeps of three different grease samples at –40°C and 25°C are shown. The storage modulus G' representing the elastic part, and the loss modulus G'' representing the viscous portion of the samples response to the oscillation as a function of the shear stress are shown.

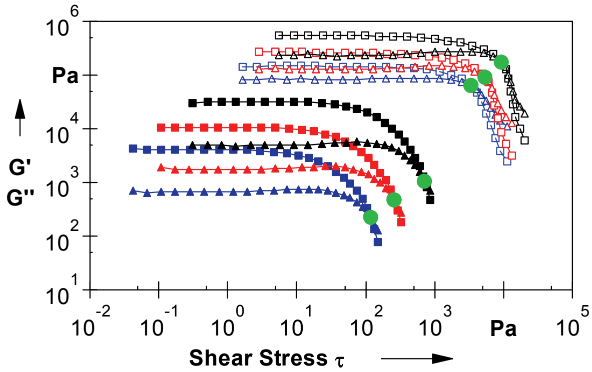

Figure 3. Strain sweeps from $\gamma = 0.001$ % up to 100 % at 25°C (full symbols) and -40°C (open symbols). Data plotted versus the shear stress. The big dots mark the respective flow points of the three greases at the two temperatures.

With the use of the tribology device and by increasing the sliding speed logarithmically, starting from small values friction factor measurements have been obtained while applying a normal load of 14 N. In Fig. 4 the friction coefficient as a function of the sliding speed for two greases measured at 25°C and -40°C are shown. For both greases, at low sliding speeds up to 5 mm/s the friction values are lower at -40°C (squares) compared to 25°C (circles), whereas at higher speed up to 100 mm/s the friction coefficient is lower at 25°C compared to -40°C. A possible explanation of these effects might be given by considering the rheology data from Fig. 3, which show that at -40°C the structure of the greases is stronger compared to 25°C. It seems that due the stronger structure and higher yield stresses a thicker film is present at lower temperatures, resulting in a smaller friction at low speeds. At higher speeds part of the grease in the contact area might be moved out and the higher yield behavior restricts a back flow of the grease. Therefore the film thickness is reduced and the friction increases.

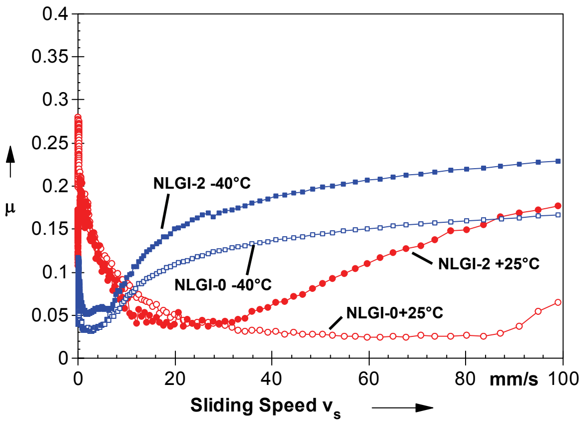

Figure 4. Friction factor vs. sliding speed for two different greases at 25°C and -40°C.

In order to investigate the static friction behavior measurements in which the friction force was logarithmically increased were performed for the same two greases. Again a normal load of 14 N was used. Before the actual measurement a run-in at a speed of 10 mm/s was performed for 10 min. followed by a rest period for another 10 min. In Fig. 5 the results of these tests are shown.

At small forces there is practically no movement and the data scatter around zero speed, represented by small values in a logarithmic scale. If the force is large enough to overcome the static friction the speed jumps from zero (or very small values) to larger values. At 25°C (circles) the friction coefficient stays more or less constant up to the maximum plotted speed. However at -40°C (squares) a step in speed can be seen as well, but at higher speeds the friction increase again. The static friction, which can be taken as the friction value at which the big jump in the speed occurs, is smaller at -40°C compared to 25°C for both greases.

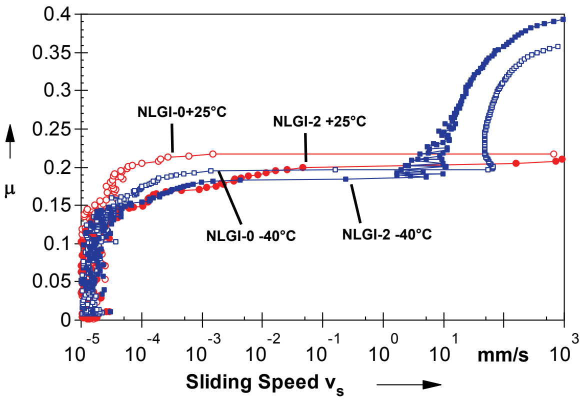

Figure 5. Static friction measurement for 2 lubrication greases at 25°C and -40°C.

Ball bearings are often lubricated with greases ensuring lowest possible friction under working conditions. Therefore a special holder for standard ball bearing has been designed and is mounted onto the tribology cell. Starting friction, running friction and roll out time after an applied speed are the most interesting parameters. The settings for the starting torque measurement are the same as for the static friction determination. The torque is logarithmically increased and the occurring speed is measured. Fig. 6 shows such starting torque measurements at -40°C, +25°C and +60°C.

Due to the influence of the yield point of the lubricant on the starting torque the ball break-off requires more force at lower temperatures. It has been shown before that the shear stress to overcome the yield point is strongly temperature dependent. The lower the temperature, the higher the yield point and the more force is required to induce flow.

The rolling friction test presented in Fig. 7 is showing a rotational speed ramp ranging from 0.1 up to 3000 rpm. The curves measured at +25°C and +60°C look similar even though the measured values are lower for +60°C. For both curves the torque

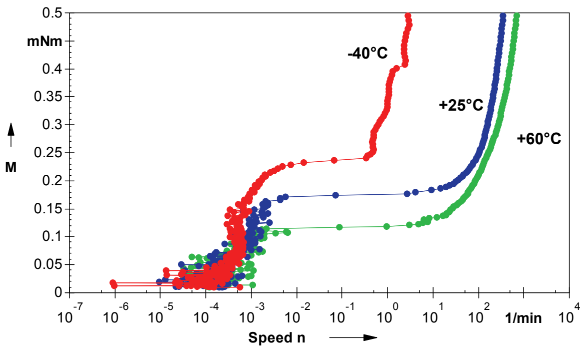

Figure 6. Starting torque measurement for a ball bearing at -40°, +25°C, and +60°C.

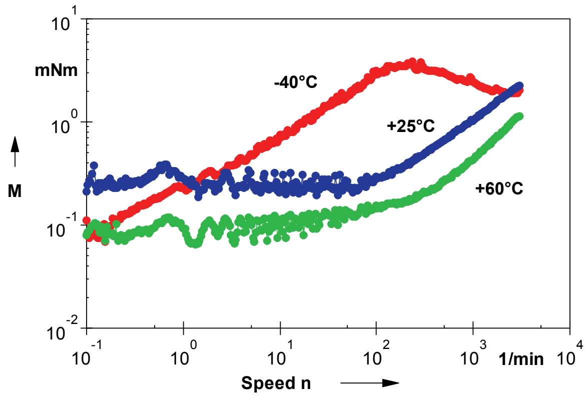

Figure 7. Speed ramp measurement for a ball bearing at -40°C, +25°C, and +60°C. Torque versus rotational speed.

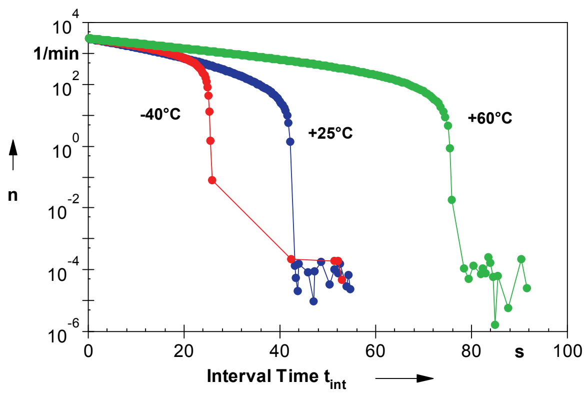

Figure 8. Roll out measurement for a ball bearing at -40°C, +25°C, and +60°C. Rotational speed as a function of the time in the roll-out interval.

slightly in the speed range form 0.1 to 100 rpm, whereas at higher speeds a steep slope can be observed indicating a lubrication problem. At -40°C the lubrication is insufficient right from the beginning since the running torque increases continuously. At 200 rpm the lubricant's viscosity drops due to friction heating resulting in an improved lubrication.

A roll-out test consists of two intervals. In the first interval the rotational speed is set to 3000rpm and held at this speed for 10 seconds, whereas in the second interval the deceleration is measured. For clarity reason in Fig. 8 only the second interval is presented. The roll out time of the ball bearing increases in the order of the temperature. At -40°C the bearing roll-out is three times shorter compared to +60°C.

MEASUREMENTS ON FOOD SAMPLES

Differentiation of Fluid Dairy Products

Various commercial fluid dairy products with varying fat amount were studied using the described tribological device. Fig. 9 shows the friction factor of these samples as a function of the sliding speed. When assessing the friction and lubrication properties of the tested dairy products, a clear discrimination between samples could be seen at several sliding speeds. The Stribeck curves generated by fluid dairy products could be divided into 3 distinct regimes, which are the boundary, mixed and hydrodynamic regime. In the low speed boundary regime, where there is virtually no pressure build-up, the friction results predominantly from the interaction of the asperities of the interacting surfaces with very small layer of lubricant. At higher sliding speeds the contact area of the two interacting surfaces are no longer in contact (hydrodynamic regime) and the friction is generated by the flow properties of the lubricant. In between these two regimes lays the mixed regime, where the contact area are in partial contact. The Stribeck curves of the fluid dairy emulsion with less than 5% fat, the mixed regime is defined by the inflection point of the Stribeck curves between a sliding speed of 3-10 mm/s. For the fluid dairy emulsion with higher fat content, there seems to be a more drawn out mixed regime between sliding speeds of 5-50 mm/s. This suggests that at low sliding speeds the sample is excluded from the contact measuring surface between the ball and the three thermoplastic elastomer plates and does not act as a lubricating agent. As the sliding speed increases the sample is adsorbed into the contact measuring surface and becomes a more effective lubricating agent.

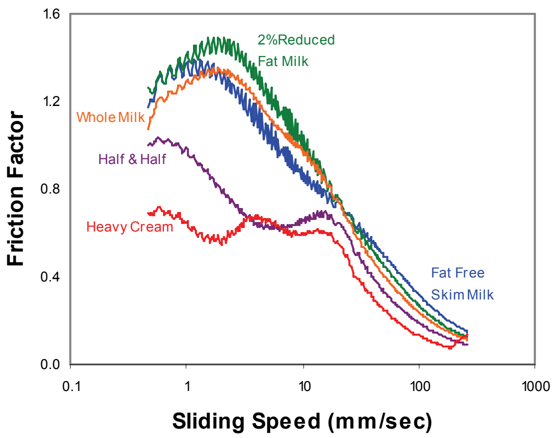

Figure 9. Friction factor as a function of sliding speed for 5 different fluid dairy samples.

The dairy samples with a fat content below 2% also have a very distinctive stick and slide pattern, which is very reproducible and seems to be a system property (as function of the sample and interacting surfaces). For whole milk and dairy products with higher fat contents we still observe the characteristic stick and slide pattern, but it is less pronounced, suggesting more lubricating action of the fat globules, due to higher fat globule population. The stick and slide pattern is visible well into the hydrodynamic regime, where the pressure build-up is enough to draw lubricant between the interacting surfaces.

Improvement of Mouthfeel of Milk

Fat-in-water dairy emulsions, thickened with maltodextrin or xanthan gum, were produced with identical micelle size distribution, apparent viscosity (20mPa.s and 70 mPa.s at 50s^{-1}) and fat volume fraction (5 vol.% and 20 vol.%) as described by Akhtar et al.[12,13].

Fat-in-water emulsions (30 vol.%, 2.8 wt.% sodium caseinate) were prepared at 50°C using a Rannie homogenizer operating at 350 bar. The hydrocolloid (maltodextrin or xanthan gum) was dissolved in demineralised water at room temperature to a solution of known concentration. Fat-in-water emulsions (30 vol.%) and hydrocolloid solution were mixed by gentle stirring in order to adjust the fat content (20 vol.% and 5 vol.%) and the viscosity. The average droplet size of the 30 vol.% emulsion was 0.86 µm and there was no change in the average droplet size when diluted to achieve 20 vol.% and 5 vol.% fat-in-water emulsions. Thermoplastic elastomer strips were cleaned with diluted soap, rinced thoroughly with tap water and dried with tissue paper by blotting. The upper ball-shaped element was made of steel. The tests were performed in duplicate or triplicate at random at a temperature of 20°C and a normal force 3 N.

The emulsions were assessed in duplicate. Panel members were asked to rate creaminess on a scale of 1 to 10, where 10 corresponds to the intensity highest rating. Principal Component Analysis was performed with CAMO Unscrambler software. The same emulsions of ~70 mPa.s and ~20 mPa.s apparent viscosities were characterised by tribology (Figs. 10 and 11) and sensory analysis (Table 1).

The sensory score increase when the apparent viscosity of the emulsion is raised from ~20 to ~70 mPa.s. This is in agreement with the results of Akhtar et al. [13]. With a background of identical droplet size distribution, fat content and apparent viscosity, the 1DE maltodextrin is far more efficient than xanthan in reducing the friction factor. Dickinson et al.[13] also found that *iso*-viscous emulsions (of identical fat content and droplet size distribution) gives a better score with maltodextrin than xanthan. They suggest that non-rheological factors may enhance the perception of creaminess.

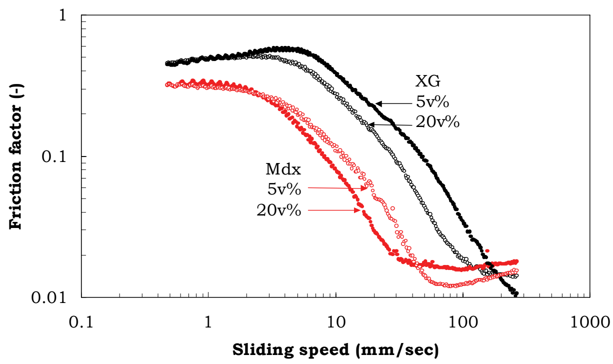

Figure 10. Stribeck curves of ~20 mPa.s emulsions.

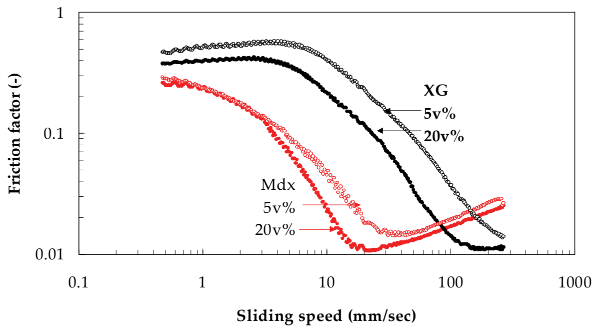

Figure 11. Stribeck curves of ~70 mPa.s emulsions.

Table 1. Summary of analytical data.

Fat (vol %)	Hydrocolloid (w%)	Visco-sity (mPa.s) at 50s^{-1}	Friction factor 2.0 mm/s	Friction factor 20 mm/s	Sensory score
5	10.8% maltodextrin	28	0.58	0.10	5
	0.14% xanthan	28	1.05	0.44	2
	14.7% maltodextrin	73	0.36	0.04	7
	0.31% xanthan	71	1.06	0.45	3
20	5.6% maltodextrin	25	0.58	0.06	6
	0.04% xanthan	19	1.02	0.29	2.5
	8.8% maltodextrin	67	0.34	0.02	7
	0.19% xanthan	81	0.82	0.23	3.5

In particular, when panelists were asked to score stickiness (or mouth-coating),maltodextrin give a significantly better score than xanthan. This work shows that beyond rheology, friction factor provides insights to explain the differences in the perception of creaminess. The Principal Component Analysis in this work reveals that the perceived creaminess is more sensitive to the friction factor than the apparent viscosity.

CONCLUSIONS

A state of the art rheometer is obviously not limited on the determination of flow behavior only. Due to the excellent speed and torque characteristics and the capability of setting and reading normal forces, it can be used for applications it was initially not designed for. This statement is underlined by the presented accessories for tribology and additional holder for ball bearings. The results obtained in the performance tests of the tribological accessory on dry and oil-lubricated friction partners were reproducible and in good agreement with data from literature. It could be shown that a single instrument can measure friction factors as function of sliding speed as well as static friction values. The temperature dependency of the starting friction, the running friction and the roll-out behavior of ball bearings were studied on a specially designed fixture mountable on the tribological accessory.

Data, obtained on food samples, showed that the friction and lubrications properties of different food samples can be assessed. Models, derived from the data appeared to be capable of predicting human sensory attributes. Tribology could be a valuable tool in assessing the mouthfeel properties associated with food systems or their components. This tool should allow food developers to better optimize the mouthfeel of fluid foods and beverages.

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