Rheometers

Rheological and tribo-rheometrical characterization of pudding

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Introduction

Both, texture, and mouthfeel, are closely related to the microstructure of foods and are therefore crucial for consumer choice and acceptability. Consequently, to enhance consumer acceptance, both formulation and production processes must be adapted to achieve the desired flow properties, texture, and mouthfeel.

Eating and swallowing are highly dynamic processes, and the oral processing undergoes various stages. The first part is dominated by the bulk properties of the sample and involves deformation and flow under shear, compression, and elongation. After that, oral processing transitions to a surface property dominated behavior, characterized by close contact interaction and lubrication of oral surfaces.[1,2]

Rheology is already a widely used technique in the chocolate and dairy industries, among others, and is employed for the characterization of the bulk property dominated regime. However, given that the oral cavity comprises of the tongue touching and sliding on the palate in the presence of a lubricating food product, tribological measurement techniques are required to quantify the frictional properties of foods. For this reason, this technique is gaining more popularity as a research tool. However, the evaluation of the obtained data is still a topic for discussion.

To showcase the use of tribology as an industrially relevant tool to quantify and compare mouthfeel, this application report focuses on the impact of different compositions of commercially available puddings on the rheological as well as tribo-rheological properties. For this, a rheometer equipped with regular and tribo-rheometrical measuring geometries is used to generate Stribeck curves and measure the respective rheological flow properties of each food product.

Materials and methods

The subject of this investigation was commercially available whole milk pudding, cream pudding, high protein pudding as well as a plant-based pudding. For this study, a Thermo Scientific™ HAAKE™ MARS™ 60 Rheometer equipped with an air-cooled Peltier temperature control module in combination with a sample hood was used. Figure 1 shows the rheometer setup.

The rheological measurements were performed using a 35 mm parallel plate geometry. Prior to starting the measurement routine, a temperature of 23 °C was set. After sample loading and trimming, a measuring gap of 1 mm was set, and the temperature was held constant for 200 s to ensure a homogenous temperature distribution within the sample. A steady state viscosity measurement was conducted in a shear rate $\dot{\gamma}$ range from 0.01 to 1000 s^{-1}. Figure 2 shows the complete HAAKE RheoWin™ Software measurement procedure.

The tribological measurements were performed using a Ball-On-Three-Discs setup. This measuring geometry, and for comparison, the human mouth as its real-world counterpart, are shown in Figure 3.

Figure 1: HAAKE MARS 60 Rheometer configuration for rheological characterization of pudding samples.

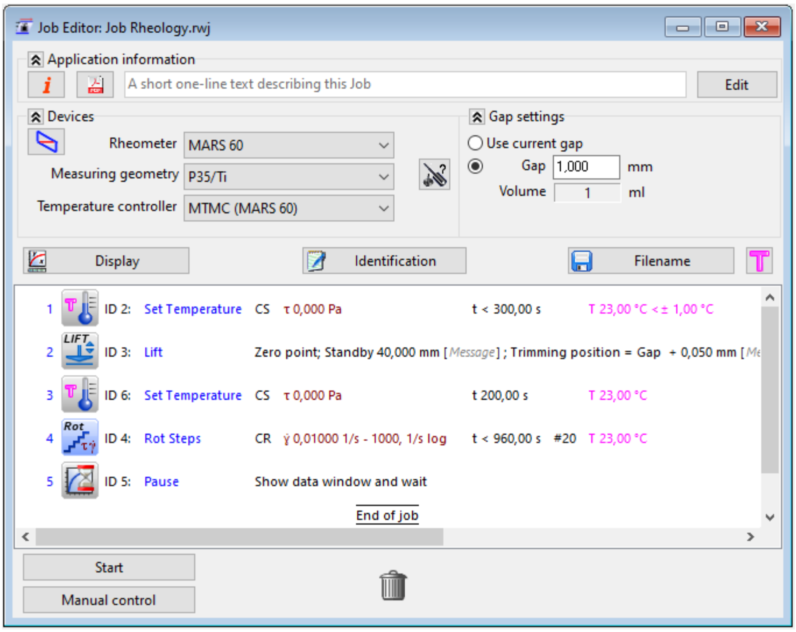

Figure 2: Measurement routine HAAKE RheoWin Software for rheological measurements.

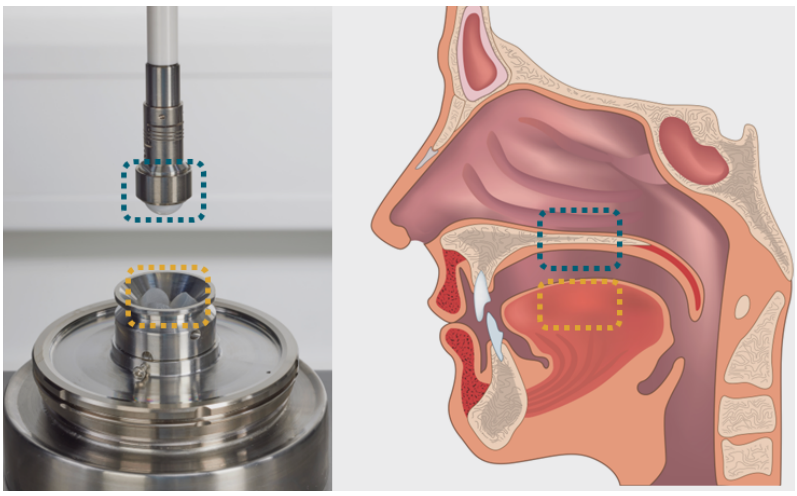

Figure 3: Tribo-rheometry measuring geometry consisting of a Ball-On-Three-Discs setup (left) as well as the corresponding tribo-system of the human mouth (tongue-yellow square, palate-blue square) (right).

To mimic the swallowing process of dairy products, a glass sphere is used to represent the palate (upper part of the tribo-pair), and Polydimethylsiloxane (PDMS) discs are used to represent the tongue (lower part of the tribo-pair).

During a tribological measurement, a body is pressed onto a surface either due to gravity or an externally applied force. As soon as the body is moved on the surface, it will experience a resistance, resulting from the friction at the interface between the body and the surface. According to this, friction can be quantified by calculating the coefficient of friction (COF) µf according to equation (1).

$$\mu f = \frac{F_f}{F_n} \qquad (1)$$

In a tribo-system, containing a tribo-pair as well as a lubricant, changes in tribological properties are commonly displayed in so-called Stribeck curves.[3] An example for such a Stribeck curve is shown in Figure 4.

At low sliding speeds vR, the surfaces of the tribo-pair are in direct contact with each other, resulting in high friction or a high coefficient of friction. This range is also commonly known as boundary lubrication. With increasing speed, lubricant is dragged between both surfaces due to hydrodynamic forces reducing the contact area and therefore the friction. Hence, the tribo-system leaves the boundary lubrication and enters the mixed lubrication regime. The more liquid is dragged between the tribo-pair, the larger the gap between them gets. As soon as the lubricant fully separates both surfaces, the friction results only from the internal friction of the lubricant itself, which corresponds to its viscosity. This state is often referred to as hydrodynamic lubrication regime.

As the viscosity of the lubricant also plays a critical role throughout the entire tribological experiment, it must be considered as well. To take this into account, a Stribeck curve also considers the lubricant's shear viscosity η as well as the applied load P, defined as normal force per contact unit length of bearing. Sliding speed, viscosity and applied load together can form the dimensionless Hersey number (He).[4]

In practice, the normal force FN instead of the applied load is often preferred for the calculation of a Hersey number.[5] Therefore, the HAAKE RheoWin Software only includes a so-called simplified Hersey number (He-sim) as described in equation (2).

$$\mathrm{He-sim} = \frac{v_R \cdot \eta}{F_n} \qquad (2)$$

Besides this, if two lubricants are characterized by similar viscosities and the applied load remains the same throughout a set of experiments, the tribological behavior can also be described by only displaying the sliding speed instead of He.

After a sufficient amount of sample was added to the PDMS pins in the lower reservoir, the rotor was lowered, and the tribo-pair brought into contact. To generate complete Stribeck curves a constant normal force of 3 N was applied and the sliding velocity of the ball was increased logarithmically from 0.1 to 600 mm/s.

During operation, the contact surfaces of a tribo-system are subject to wear, especially at the beginning of operation. The transient character of this so-called running-in, derives from the wear down of microscopic surface asperities which results in a reduction of friction over time. This phenomenon can usually be observed during the first measurement runs. To obtain reproducible measurement data it is therefore necessary, to perform the same experiment several times using the same tribo-pair while reloading fresh lubricating sample after each run and thoroughly cleaning the surfaces with isopropyl alcohol before. Once the tribological data does not change significantly anymore and the curves overlay, the end of the running-in period has been reached. This is usually the case after 2–3 runs. Hence, in this study the 3rd repetition run was used for further evaluation. Figure 5 shows the HAAKE RheoWin Software measurement procedure used for the tribological measurements.

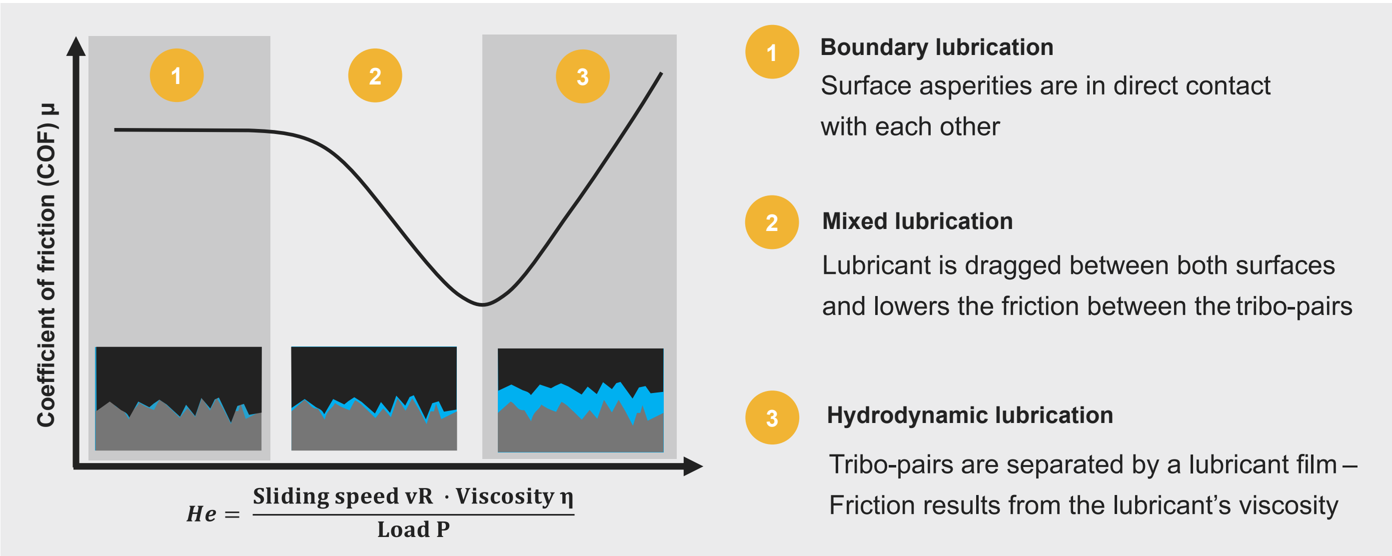

Figure 4: Example of a common Stribeck curve.

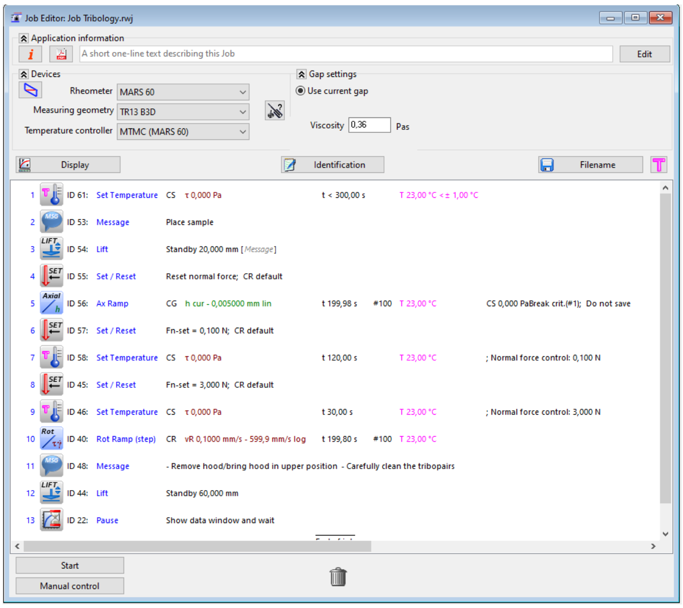

Figure 5: Measurement routine in HAAKE RheoWin Software for tribological measurements.

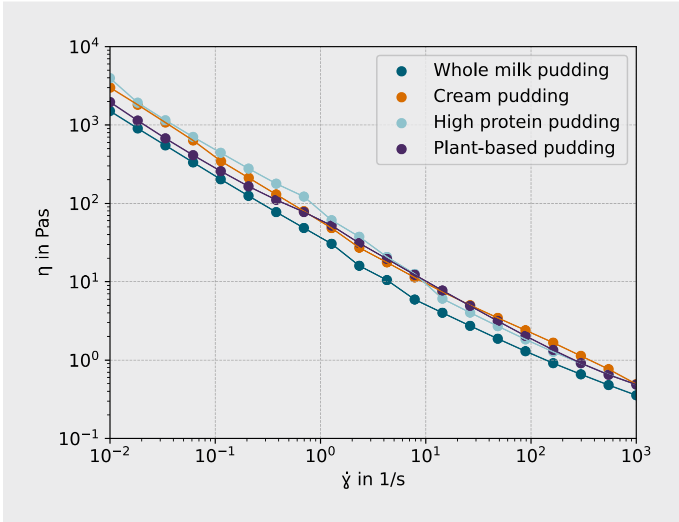

Figure 6: Comparison of viscosity curves of different commercially available types of pudding.

Results & discussion

Figure 6 shows the viscosity curves of the different puddings investigated in this study, representing their flow properties. All four samples show a decreasing shear viscosity with increasing shear rate and can therefore be classified as non-Newtonian materials. As the actual differences regarding the shear viscosity η are minor, all pudding samples behaved rather similarly regarding their flow properties.

Since many materials, including dairy samples, show a non-Newtonian behavior with a shear rate dependent viscosity, usually a high-shear viscosity η_{1000} at a shear rate of 1000 1/s is chosen for the creation of Stribeck curves when samples with different viscosities are compared.[1] To follow this guideline, Table 1 illustrates η_{1000} for each pudding.

Sample	η_{1000} / Pas
Whole milk pudding	0.36
Cream pudding	0.49
High protein pudding	0.48
Plant-based pudding	0.49

Table 1: High shear viscosities of the different pudding samples.

By applying the concept of the simplified Hersey number, the tribological properties of all screened substances can be identified depending on their Stribeck curves illustrated in Figure 7. To correlate the effect of the different nutritional composition on the friction properties, a corresponding bar plot is displayed as well.

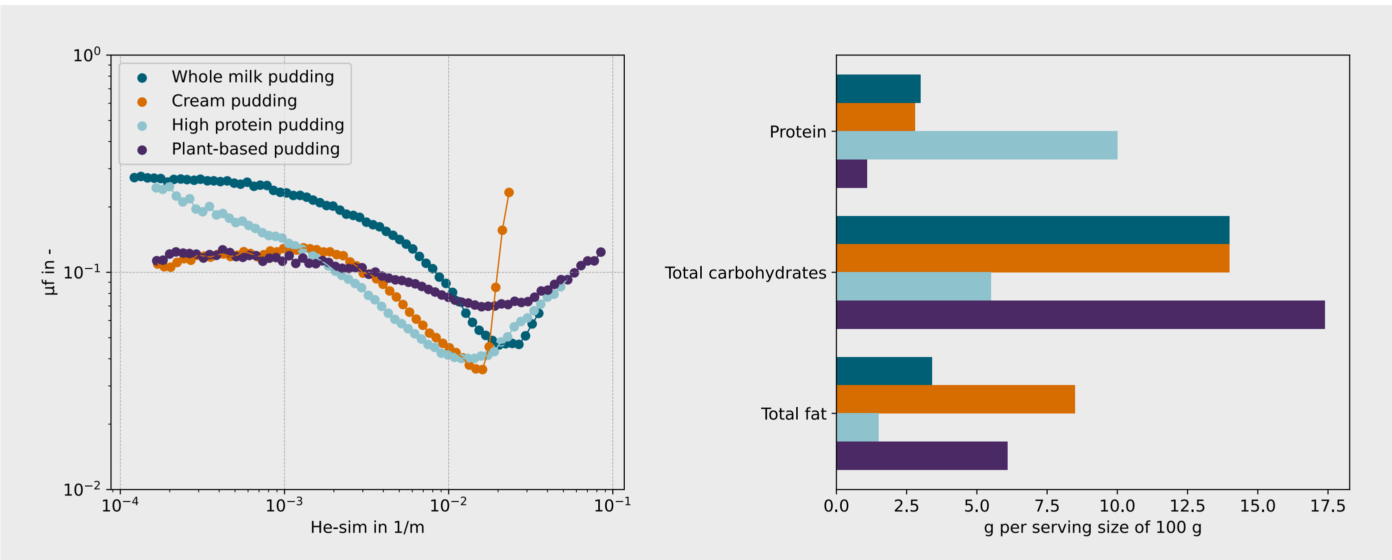

Figure 7: Tribological data as well as nutrition facts of the pudding types used in this study.

In contrast to the shear viscosity curves presented in Figure 6, the frictional behavior of the screened substances is much more diverse. This is mostly related to the differences in protein, total carbohydrates, and total fat content. Depending on the type and structure of these macromolecules, different bio-lubrication mechanisms can be present. A brief overview of these mechanisms is shown in Figure 8.

The total fat content of the cream pudding (8.5 g/100 g) and the plant-based pudding (6.1 g/100 g) are higher compared to the whole milk pudding (3.4 g/100 g) and the high protein pudding (1.5 g/100 g). Fat crystals can act like rolling balls in a ball bearing to increase lubrication. This results in an overall lower friction in the boundary lubrication regime of the cream pudding as well as the plant-based pudding below simplified Hersey numbers of 10^{-3} 1/m. However, despite the higher total fat content of the cream pudding, μf does not differ significantly from the plant-based pudding in the boundary lubrication regime.

Contrary to this, the friction properties of both samples differ in the mixed as well as the hydrodynamic lubrication regime. As the hydrodynamic lubrication regime is mostly influenced by the samples' viscosity and the different pudding types show similar flow properties according to Figure 6, it can be assumed that in the case of the cream pudding most of the sample is dragged out of the gap between the two rotating bodies due to centrifugal forces at very high rotational speeds. This leads to a drastically increasing friction within the tribo-system. Due to the higher shear at larger sliding speeds, a lot of energy is introduced into the surface boundary of the tribo-system. This results in coalescence of oil droplets and consequently, the formation of a lubricating oil film. Hence, a friction reduction in the mixed lubrication regime can be observed. However, the plant-based pudding shows a less pronounced reduction in friction during the transition from boundary to mixed lubrication. According to the list of ingredients, coconut fat was used for this product. Therefore, it is likely that the coconut fat lubricates the sliding bodies much less compared to the animal-based fat of the other products.

Interestingly, the high protein pudding shows no boundary friction plateau in contrast to the other puddings. This could be related to the shape of the macromolecules. In tribological contacts non-spherical protein molecules slide, whereas their globular counterparts roll. Besides this, the formation of polysaccharide-protein complexes could also affect the friction behavior.

Although the whole milk can be characterized by having a low protein content as well as the lowest fat content, it shows the highest overall friction. Additionally, due to the low protein content, the formation of a polysaccharide-protein complex to enable complex lubrication is not likely, which may explain the higher overall friction.

Tribology offers a lot of different possibilities for evaluating the lubrication behavior foods. However, for a correct interpretation, additional characterization techniques like the use of quartz crystal microbalances are necessary to link the adsorption behavior of the different macromolecules on the model surfaces to the associated lubrication mechanisms.[8]

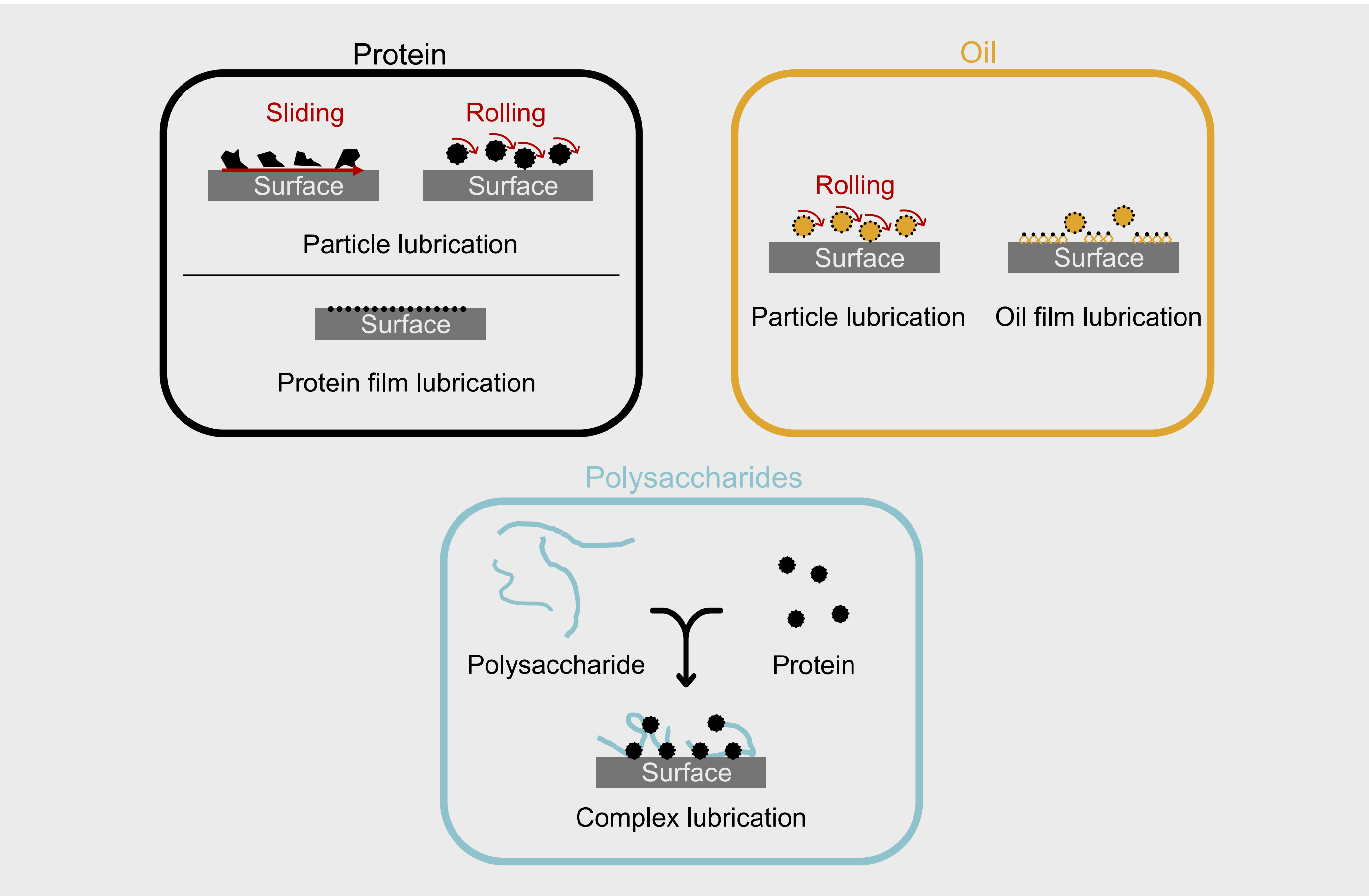

Figure 8: Bio-lubrication mechanisms of protein, oil as well as polysaccharide according to [6, 7].

Conclusion

In this application report, various types of pudding were evaluated in terms of their flow as well as their lubrication properties. Despite only a slight variation in flow properties, all food products showed considerable differences in their frictional properties. These differences can be linked to their respective nutritional values as protein, fat and carbohydrate content can influence the lubrication in the tribo-system 'glass sphere—PDMS discs'. Based on such findings, the influence of microstructures of, for example, plant-based food products can be compared to their dairy-based counterparts, speeding up both, product improvement and new formulations, to meet consumer expectations.

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