Oral texture perception of semisolid foods in relation to physicochemical properties

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ORAL TEXTURE PERCEPTION OF SEMISOLID FOODS IN RELATION TO PHYSICOCHEMICAL PROPERTIES

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To produce healthy foods that we like to eat, it is essential to understand how sensory perception of food relates to food properties. The work in this thesis focuses on understanding oral texture perception of semisolid foods, in particular the sensory property creaminess. This thesis describes the attempts to identify physicochemical properties underlying oral texture perception, as well as to find instrumental measurements other than bulk shear rheometry that can measure these properties. The results increase understanding of the mechanisms behind the perception of orally perceived texture attributes, provide instrumental methods to measure specific attributes, and give insight in the oral processing conditions during perception. The studies used mayonnaises, and in some cases also custards, to represent semisolid food products. The instrumental measurements included different types of rheological measurements in shear, operating at small and at large deformation conditions. The relations of oral texture perception with these measurements were compared to relations with rheological measurements in elongational flow and relations with a set of novel instrumental measurements on nonrheological properties (e.g., friction) and/or under oral-like conditions (e.g., the effect of saliva). Relations were established by modeling, univariate correlations and multivariate predictions.

The results showed that rheological measurements in shear are very useful instrumental techniques to measure oral texture perception of mayonnaises. Creaminess and most other texture attributes could be predicted well by parameters from shear rheological measurements alone. For a complete prediction of texture attributes, additional properties are required, but these were not obtained by the other measurement techniques tested in this thesis. Addition of parameters reflecting the effect of saliva improved the predictions only to some extent. Attributes perceived after swallowing benefited more from the addition of these extra parameters than mouthfeel attributes, perceived during mastication. Replacement of rheological measurements in shear by rheological measurements under partial elongation or addition of friction measurements improved the predictions of the main attributes of mayonnaises only to a limited extent. It is remarkable that the complex mechanisms of oral processing and texture perception can be represented relatively well by one single type of properties, reflecting behavior in deformation, which can be determined by conventional measurements.

The relations between perceived texture and parameters from shear rheological measurements show that the characteristics of mayonnaises at the transition from solid-like to liquid-like behavior are most important for the perception of most texture attributes. A mayonnaise is perceived as creamy when this transition from rest to flow happens suddenly and rapidly. These characteristics are expressed by tan δ at high oscillating strain rates in a dynamic stress sweep measurement. Measurements in large deformation, i.e., the steady shear-rate measurement and the flow-curve measurement are also relevant in relation to texture perception. A creamy mayonnaise shows a limited decrease in viscosity in large deformation flow. Thickness mouthfeel of mayonnaises could be predicted well by a relative simple physical-physiological model that predicts the shear stress perceived on the tongue at simplified oral processing conditions.

The results indicate that the mouth senses the rheological behavior of the food bolus under oral deformation and translates this into perception of texture attributes. All mouthfeel attributes, including those perceived immediately after food intake, require a certain minimal amount of deformation, so there are no relations with rheological properties measured at rest. The level of oral deformation consists of deformation conditions at which the structure is disrupted and the mayonnaises start to flow as well as of conditions of large deformation at very high shear rates. Elongational deformation and the compression movement are included in oral processing but play a minor role in perception compared to shear deformation. Friction properties and the effect of saliva on properties of the food bolus and oral coating are also sensed and play primarily a role for textural afterfeel attributes, because oral deformation is minimal during perception of these attributes

Comparing the results of mayonnaises with those of custards showed that there are distinct differences in perception within the group of semisolid foods. The differences in composition and structure between custards and mayonnaises resulted in very different sensory perceptions and different relations of texture attributes with physicochemical properties. For custards, the saliva-induced breakdown of starch, absent in rheological measurements, is more essential than it is for mayonnaises.

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INTRODUCTION

Background and Aim of this Thesis

Consumption of food products plays an important role in our daily life. Eating is not only vital for our well-being, but can also be a very pleasant activity, especially when we like the food we eat. Food liking depends to a large extent on the sensory properties of the food; the way the food looks, smells, tastes and feels. While eating, the perception of these sensory properties is integrated in our brain with more subconscious factors such as expectations and state of mind. This results in a conscious sensory perception of the food ("Wow, this is creamy!") as well as an opinion on food liking ("This is delicious, give me more!").

Perception of the sensory properties of food starts when we see the food and pick it up. A certain bite size is taken into the mouth, where we chew it and mix it with saliva, and finally swallow it. Even after the food sample has left our mouth, we can still perceive some of its sensory properties. The origin of sensory properties lies in the structures and accompanying physicochemical properties of the food products; a salty taste originates from the presence of salt molecules, a crispy feeling from the physical breaking properties, a blue color from the wavelength-reflecting properties of the structure, a rotten-egg smell from a concentration of certain odor molecules, and the feeling of thinness from the viscosity of the product. During handling and mastication, the structure and physicochemical properties of the food change; a piece of chocolate melts in the mouth, a cookie is broken down and gets soggy, and a pudding is broken down into lumps. All these structural and physicochemical properties and their changes are sensed by our senses, such as sight, hearing, taste, smell and touch, and translated into sensory properties. To get more insight in the way in which perception of sensory properties works, it is important to identify the structural and physicochemical properties underlying the different sensory properties.

Until recently, much of the research on the physicochemical properties underlying sensory perception has been done for taste, smell and odor properties. Research on the origins of texture and mouthfeel was limited to properties such as hardness and brittleness of solid foods and to perceived thickness of (semi)liquid foods. The work in this thesis focuses on oral texture perception of semisolid foods, in particular on the sensory property creaminess. This work was part of a large multidisciplinary project of the Wageningen Centre of Food Sciences. In this project, called 'Physical and chemical aspects of sensory attributes', scientists from sensory science, oral physiology and material science cooperated to try to unravel the relations between texture perception, food properties and the mastication process. Food liking and subconscious processes in the brain were outside the scope of this project. Also properties perceived outside the mouth, such as color and thickness perceived by hand, and properties associated with foods other than semisolids were not investigated. The aim of the work in this thesis is to identify physicochemical properties underlying oral texture perception of semisolid foods, in particular creaminess, as well as to find instrumental measurements other than bulk shear rheometry that can measure these properties. With these results, origins of oral texture properties and the relevance of specific instrumental measurements are established, as well as oral mastication conditions during perception. This will increase understanding of the mechanisms of oral texture perception. This is essential when we want to produce healthy foods that we like to eat. Furthermore, it will provide instrumental methods to determine specific sensory properties. This enables industry and researchers to perform quick screening of samples without the use of time-consuming and expensive sensory panels.

To achieve this aim, semisolid food products were subjected to assessments by sensory panels and to instrumental measurements. Mayonnaises, and in some cases also custards, were used to represent semisolid food products. The products were bought in local supermarkets (commercial products) or produced within the project according to recipes and processing conditions with specific variations (model products). The instrumental measurements included different types of rheological measurements in shear, operating at small and large deformation conditions. In addition, rheological measurements in elongational flow were applied, as well as a set of novel instrumental measurements on nonrheological properties and/or operating under more mouth-like conditions. Relations between sensory properties and physicochemical properties were established by modeling, univariate correlations and multivariate predictions. To interpret these relations, also relations among the sensory attributes and with ingredients were assessed.

Semisolid Food Products

Semisolid food products make up a large part of the food we eat. These products show behavior in between that of a true solid (which breaks) and a true liquid (which flows). For this reason, they are also called semiliquid products. The consistency of a semisolid product can vary between soft solid (e.g., margarine) and viscous liquid (e.g., yoghurt). Their behavior is often very complex, because they have solid-like as well as liquid-like properties. It is primarily determined by the composition and structure of the foods. In this thesis, two different semisolid food products are investigated: mayonnaises and custards.

Mayonnaises

Mayonnaises are products that contain a relative large amount of fat. The structure consists of fat droplets dispersed in a continuous water phase. This structure is called an oil-in-water emulsion. Traditionally, a true mayonnaise contains 70% to 80% of fat (Depree & Savage 2001). At these high fat contents, the fat droplets are closely packed together. Products with lower fat contents are officially called dressings. In the studies of this thesis, all mayonnaise-like products are called (full-fat or low-fat) mayonnaises, whatever the fat content. Mayonnaises in Dutch supermarkets contain between 10 and 80% of fat. When the amount of fat is lowered, the mayonnaise becomes thinner. To overcome this, thickening agents such as (modified) starch, xanthan, and guar are added to the water phase. Lower fat levels require more of these thickeners. Other important ingredients are egg yolk to emulsify the fat droplets, vinegar to acidify and sugar to sweeten the product.

In real life, mayonnaise is rarely eaten on its own, rather together with a solid (e.g., potato fries). In the studies of this thesis, however, panelists tasted the mayonnaise samples always as such, as we wanted them to characterize the mayonnaises themselves.

Custards

Custard ('vla') is a popular, typical Dutch dairy product, which is eaten as sweet dessert (Rosenberg 1990). Custards are available in many different flavors, but the most basic one, vanilla-flavored, are used in the studies of this thesis. At the present day, the product is composed of milk, (modified) starch, carrageenan, sugar and vanilla flavor. Fat contents can range from 0.1 to 3.5%. The structure of custard is an oil-in-water emulsion, but the dispersed phase of fat droplets plays a limited role compared to mayonnaises, due to the low amount of fat. The thickeners in the water phase (starch and carrageenan) are much more important for its structure. The extent to which the starch granules in custards are dissolved (gelatinized) plays an important role in the behavior and properties of custards (Janssen *et al*. 2007).

Eating of Semisolid Foods: Oral Processing and Sensory Perception

When a food product is eaten, a portion is taken into the mouth and subjected to oral processing. The term 'oral processing' describes all the processes that occur in the mouth between ingestion and swallowing, such as chewing and mixing with saliva. The aim of oral processing is to break up the product, neutralize its pH, and form one smooth, fairly homogeneous substance that is safe to swallow. The substance formed by the processed food and saliva is called the food bolus. After swallowing, a layer of this substance is retained in the mouth and covers the oral tissue of tongue and cheeks. This layer is called the oral coating. During oral processing and after swallowing, the properties of the food bolus and oral coating are sensed by several types of sensors in the mouth and nose and translated into a sensory perception of the food product.

Oral processing

In case of semisolid foods, a bite of food is easily formed into a smooth, homogeneous food bolus. Oral processing of this semisolid food bolus is a combination of oral movements by tongue and jaws, mixing with, and acting of saliva, and warming to oral temperatures. The oral movements are primarily confined to those between the tongue and palate, because most humans (Engelen 2004) do not apply their teeth to chew on semisolids. The tongue describes a series of movements, including compression and decompression to and from the palate and lateral movements from front to back and from side to side. A specific wedge-like movement is required to swallow the food bolus. All these oral movements result in shear as well as elongational deformation of the food bolus (de Bruijne 1993; Nicosia & Robbins 2001; van Vliet 2002). The exact pattern of forces and velocities caused by the oral movements is still largely unknown. A pioneering study of Shama & Sherman (1973a) showed that not only the amount, but also the type of oral deformation varies for different semisolid food products. Thin food products (milk) seem to be subjected to deformation at constant force conditions, and the resulting strain (rates) sensed. Thicker semisolid foods (peanut butter) seem to be subjected to constant strain rates, and the resulting forces sensed. The consistency of mayonnaises and custards suggests that they are deformed at conditions in between the two cases described above, varying in force as well as strain (rate). A general strain rate that can represent the deformation rate during oral processing of semisolid foods ('the effective oral shear rate') is still under discussion (Wood 1968; Cutler *et al*. 1983; Richardson *et al*. 1989; Stanley & Taylor 1993; Houska *et al*. 1998), but values range in most studies between 10 and 50 s^{-1}. Other research has shown that the maximum force that can be obtained by the movement of the tongue can be estimated at 50 Pa (de Bruijne *et al*. 1993).

These oral forces and velocities result in mechanical deformation and structure breakdown of the semisolid food. The structure of the food is further affected by mixing with saliva. The role of saliva in oral processing is multifold: it decreases the viscosity of the food bolus by dilution and possibly by enzymatic breakdown by α-amylase when there is starch present, it acts as a lubricant for the bolus because of its large salivary proteins, and it neutralizes the impact of certain tastants such as acid (Engelen 2004). Research has shown that the enzymatic breakdown of saliva occurs fast enough to affect the properties of the food while it is still in the mouth (de Wijk *et al*. 2004). As the outer layers of the food bolus are at an earlier stage and to a larger extent affected by saliva than the inside, the food bolus is not really homogeneous (Janssen *et al*. 2007).

Oral sensory perception

The result of oral processing is that initial structure and properties of the food (bolus) changes. These properties and their changes over oral processing time are sensed by several types of sensors in the mouth and nose, such as taste cells, olfactory receptors, mechanoreceptors, and thermoreceptors. The signals of these sensors are integrated by the brain into a conscious sensory perception. The resulting orally perceived properties, also called sensations or attributes, are in many cases based on a combination of physicochemical properties. Examples of sensory attributes of semisolid foods are thickness, vanilla flavor, prickling and creaminess. The different orally perceived attributes can be classified according to their receptors and time of perception. Odor attributes (e.g., vanilla odor) are smelled at the start of and after oral processing by the olfactory system. Flavors and tastants (e.g., off flavor, salt) are perceived during oral processing by the olfactory system and by taste cells in the mouth. Trigeminal attributes (e.g., prickling) are perceived by pain receptors and texture attributes (e.g., roughness) primarily by mechanoreceptors during and after oral processing. These latter groups of attributes can be perceived during oral processing by the properties of the food bolus (i.e., mouthfeel attributes) or after swallowing by the properties of the oral coating (i.e., afterfeel attributes).

Oral perception of texture is the focus of this thesis. Texture can be defined as the sensory and functional manifestation of the structural, mechanical and surface properties of foods, detected through the senses of vision, hearing, touch and kinesthetics (Szczesniak 2002). Some of the texture attributes are associated to bulk properties, involving the whole sample (e.g., thickness). Others are associated to surface properties, involving the outer layer of the food bolus only (e.g., roughness), or associated to both types of properties (e.g., creaminess) (de Wijk *et al.* 2006a). Some texture attributes are related to characteristics of the fat in the product (e.g., fattiness), to characteristics of the continuous thickened water phase (e.g., powderyness), or to both.

Creaminess is a very special texture attribute (Howe 1996; Frøst & Janhøj 2007; Tournier *et al*. 2007). It is typically associated with the consumption of semisolid foods. Creaminess is an important attribute, as it is highly appreciated by consumers. It can be perceived as a mouthfeel and as an afterfeel. Panelists describe creaminess as a full, velvety feeling in the mouth (Weenen *et al.* 2003). Kokini & Cussler (1983) and others (Elmore *et al*. 1999; de Wijk *et al*. 2006a) showed that creaminess mouthfeel can be predicted from a combination of perceived thickness and perceived smoothness. Flavors can also play a role in the perception of creaminess. The origins of creaminess seem multifold, bulk- as well as surface-related and affected by fat as well as thickener ingredients. Creaminess is therefore very complex to understand (Szczesniak 2002).

Table 1.1 Mouthfeel and afterfeel attributes of texture as studied in this thesis, including their definitions by the panel members.

Attribute	Definition by panel members
Thick mouthfeel	The thickness of the product in the mouth after taking a bite. This attribute is perceived by moving the tongue up and down against the palate. Moving up and down is important, especially for thicker products. When a product is very thin, it spreads directly throughout the whole mouth.
Airy mouthfeel	The degree to which the product is airy. The product feels light on the tongue, and is a bit foamy, and it takes little effort to disintegrate. Disintegration is executed by the tongue. Whipped egg white is very airy (maximum score) and not coherent; it takes little effort to disintegrate. Airy is perceived between tongue and palate.
Grainy mouthfeel	The degree to which the product, regarding structure, contains a few or a lot of grains. It is judged by rubbing the tongue against the palate.
Creamy mouthfeel	It is a soft, full feeling in the mouth, as well as it is somewhat 'lobbig', supple, and it is soft. The product is neither rough nor dry. It leaves a soft, fatty feeling. It is often a combination with fat. It is a feeling that is perceived in the whole mouth and with the whole inside of the mouth. There is a velvety feeling in the whole mouth.
Sticky mouthfeel	Sticky feeling that can be perceived by tongue and palate. A really very sticky product, for instance, is the caramel of a candy bar. It is perceived between the teeth during mastication. The degree to which the product comes loose from different parts of the mouth determines the intensity of sticky.
Heterogeneity mouthfeel	The feeling you get when a product has at the same time something thin and something thick. This feeling can also be described as cloudy or flocky. It arises when the product does not melt evenly, after putting it in the mouth. Sometimes, it takes a while before the feeling is perceived. This feeling is perceived in the whole mouth.
Dry-mealy mouthfeel	Represents a dry/mealy feeling in the mouth. Difficult to swallow. It is perceived between tongue and palate. It is difficult to remove the product and it extracts saliva from the mouth. When moving the tongue through the mouth, it feels rough. You are inclined to have a drink.
Melting mouthfeel	The speed with which the product becomes liquid and spreads in the mouth. It is the gradual process of becoming thinner in the mouth. The product is also diluted with saliva and it is a term that is measured in time. The feeling is perceived between the tongue and the front of the palate.
Fatty mouthfeel	Represents a fatty feeling in the mouth. Indicates a slippery feeling and a sealed coating on the palate. At first, it is perceived on the inside of the edges of the teeth, later also on the outside of the edges of the teeth.
Powdery mouthfeel	The product has a structure of meal porridge and it homogeneous. The powdery feeling can be perceived between tongue and palate.
Creamy afterfeel	The degree to which the product leaves a soft/creamy feeling in the mouth. It is related to the creamy mouthfeel sensation, but the soft/creamy sensation is less intense than with creamy mouthfeel (velvety feeling).
Sticky afterfeel	Sticky feeling on teeth and molars after expectorating the product. The degree in which the product leaves a sticky feeling in the whole mouth.
Fat afterfeel	The degree to which the product leaves a fatty feeling. It is a typical coating of the mouth. It can be perceived on teeth and palate by sweeping it with the tongue.
Slimy afterfeel	Degree to which the product leaves a slimy feeling after swallowing. One is inclined to keep on swallowing. It is made up from thicky, stringy saliva, sticking together.

Sensory Measurements

To study texture perception, the texture properties of the food products have to be characterized first. Three types of sensory characterization tests can be performed: a discrimination test that determines whether a product is different from another; an acceptance test that determines the liking and acceptance of a product; and a descriptive analysis test that determines the intensity of specific attributes (Stone & Sidel 1985). The latter test is the most sophisticated, as it provides a complete quantitative sensory description of a set of products. A specific version of this test, called quantitative descriptive analysis (QDA, Stone & Sidel 1985) is applied in the studies of this thesis.

Assessments according to quantitative descriptive analysis by a group of panelists start with sessions in which the panel members describe the different attributes that can be perceived by tasting the set of studied food products. This list of attributes is adjusted by discussions and retasting until a set of non-overlapping attributes is left and all panel members agree on the definition of every attribute. Panel members are then extensively trained with samples similar to the ones that are to be tested. Finally, samples are tested in randomly order and in replicate by every panel member, where they rate the intensity of every attribute on a scale of 0 to 100. Table 1.1 shows the list of texture attributes assessed in the sensory measurements of this thesis.

The judgment of food products by panel members in descriptive analysis is different from judgments by consumers; not only because the panel members have received extensive training, but also because they are only allowed to rate the intensity of the attributes and not the overall liking or acceptance of the samples.

Instrumental Measurements of Physicochemical Properties

Physicochemical measurements determine properties that have a physical and/or chemical background. Examples are density, conductivity, breaking force, friction, viscosity, and elasticity. Properties and measurements are called fundamental when the measured properties are independent of the instrument and the measurement conditions are well defined. These fundamental properties only reflect the behavior of the sample, hence they are called material properties. In empirical measurements, the measured properties are more subjective, as they depend on ill-defined measurement conditions; for example, the breaking force depending on different shaped samples. Results from empirical measurements should be regarded more qualitative than quantitative. Empirical measurements can be very useful, though as they can give an indication of sample behavior in situations that are too complicated to capture with fundamental measurements. Measurements operating at mouth-like conditions are often more empirical. Physicochemical properties can also be divided according to bulk properties (e.g., density) and surface properties (e.g., friction).

In the studies of this thesis, most of the physicochemical characterization concern rheological properties. Most of the conventional rheological measurement techniques for semisolid products determine bulk rheological properties and can be considered largely fundamental.

Rheological properties

Rheological properties are properties associated with deformation and flow of materials. They reflect the manner in which materials respond to applied stress (which is similar to force) or strain (Steffe 1996). The stress or strain of deformation can be applied in a lateral movement (shear deformation) or in a compression or extensional movement (elongational deformation), see Fig. 1.1. Rheological measurements can be performed on solid materials (determining the force required to bend or break it) as well as on liquid materials (determining the force required to stir it). Solid materials (rubber band, gelatin pudding) show elastic behavior when they are deformed; after small deformation, they return to their original shapes. At larger deformation they will break or rupture. Liquid materials (water) show viscous behavior when they are deformed; they start to flow and do not return to their initial shapes. Viscosity is an important property for liquids. Semisolid materials show both types of behavior, depending on the exact conditions of deformation (such as amount and timescale). Their behavior is called viscoelastic.

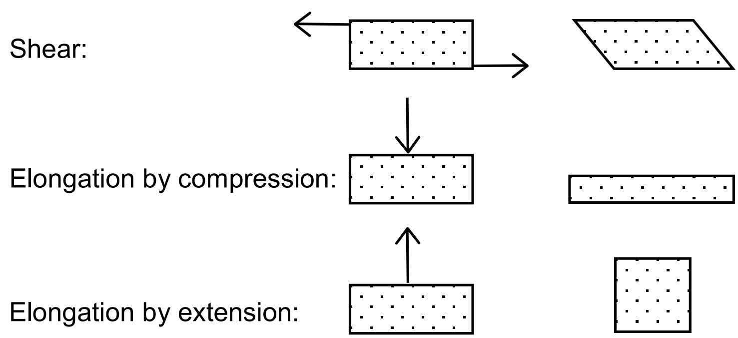


Figure 1.1 Deformation in shear and compression/extension of a sample.

Bulk rheometry in shear

For semisolid materials, shear rheometry is more common than rheometry in elongation. The rheometers used for the shear rheological measurements in this thesis are rotational viscometers. These can be operated at controlled-stress or controlled-strain conditions. In case of controlled-stress conditions, a certain stress is imposed on the sample, and the resulting strain measured. For controlled-strain conditions it is vice versa. The measurements can be divided in small deformation measurements and large deformation measurements. Small deformation measurements only deform the sample to a limited extent. Because of this limited amount of deformation, the viscoelastic semisolid sample behaves more as a solid material than as a liquid material. This makes it possible to measure the elastic properties of the visco-elastic material. At larger deformation, the solid-like structure of the visco-elastic sample breaks down and the material starts to flow. The material behaves more as a liquid and viscous properties are primarily determined.

Small deformation rheological measurements
Small deformation measurements determine initial rheological properties, measured on undisturbed samples, which are still 'at rest'. To create sufficient time for determining the initial rheological properties within the limited time of deformation, the deformation movement is applied in a dynamic i.e., oscillating way; the sample is deformed to a small extent alternating in one and in the opposite direction. The oscillating values of stress and strain allow the determination of two types of properties: the storage modulus (G'), reflecting elastic/solid-like behavior and the loss modulus (G''), reflecting viscous/liquid-like behavior. A third property, tan δ, is calculated as the ratio of G'' and G'.

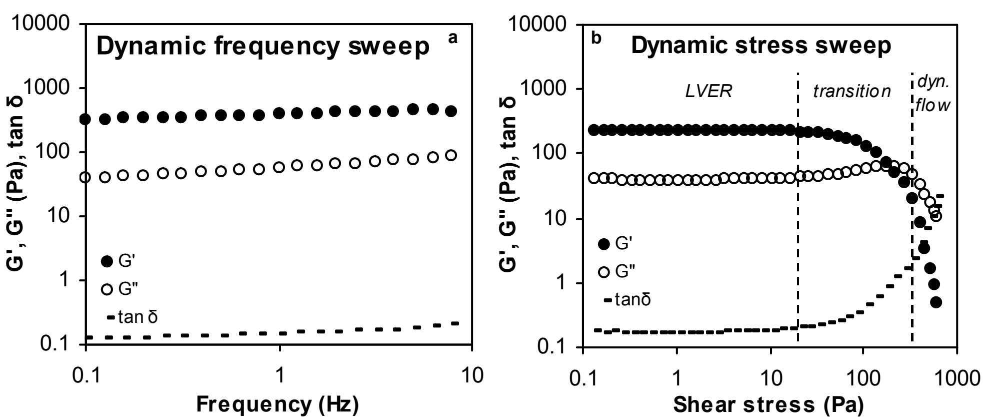


Figure 1.2a,b Example of (a) a dynamic frequency sweep and (b) a dynamic stress sweep measurement.

In this thesis, two types of small deformation measurements are applied: dynamic frequency sweeps and dynamic stress sweeps. In a dynamic frequency sweep, the storage modulus, loss modulus and tan δ are measured at increasing frequencies (see Fig. 1.2a). The oscillating hin-and-forth movement is performed at a constant imposed stress, but at increasing speeds. Fig. 1.2a shows that the storage modulus is larger than the loss modulus for all frequencies. This indicates that elastic behavior is more important than the viscous behavior. As expected, the sample is 'undisturbed' and behaves as a viscoelastic solid.

In a dynamic stress sweep, the frequency of the oscillating movement is kept constant and the imposed stress increased. It measures the behavior of the storage modulus, loss modulus and tan δ as a function of stress increase. Three different regimes can be distinguished in this measurement (see Fig. 1.2b): at low, mediate, and high stresses. At low stresses (left-side of Fig. 1.2b), the behavior is similar to that measured by the dynamic frequency sweep; the storage modulus is larger than the loss modulus, and the sample behaves as an undisturbed viscoelastic solid. This part of the curve is called the Linear ViscoElastic Region (LVER), because both moduli stay constant when stress increases. When the stress of the oscillation is further increased, deformation is no longer small and the structure of the sample starts to break down. At high stresses (right-side of Fig. 1.2b), the sample has become more liquid-like, and the storage modulus has declined strongly and is smaller than the loss modulus. The sample behaves as a viscoelastic liquid. The region in between, at mediate stresses, reflects the transition between both behaviors including the end of the LVER. Dynamic stress sweep measurements are applied to characterize the changes from initial to fluid structure and properties.

Large deformation rheological measurements
In large deformation measurements, a large, continuous deformation is applied to the sample to measure the flowing properties of the sample, that is, its viscosity. The sample behaves liquid-like. Only at the beginning of the measurement, when the sample has to start flowing, elastic behavior can sometimes be measured. This is then called the transient regime. Most conventional large deformation measurements apply a certain strain rate and measure the stress required to achieve this strain rate. The apparent viscosity of a sample is calculated as the ratio of the stress and the strain rate. In this thesis, two types of large deformation measurements are applied: steady shear-rate measurements and flow-curve measurements.

In a steady shear-rate measurement, a constant strain rate (the shear rate) is applied to the sample and the resulting stress and viscosity determined in time (see Fig 1.3a). This set-up can be compared to stirring at a constant speed. The measurement is applied to show the time-dependent rheological properties. Most semisolid foods are (pseudo-) thixotropic. This means that the viscosity decreases during deformation at constant shear rate, see the right side of the curve in Fig. 1.3a. This is caused by breakdown of the structure. A true thixotropic sample regains its initial structure and viscosity after some time of rest. Most of the semisolids foods do not recover completely, hence they are called pseudo-thixotropic. The left side of the curve in Fig. 1.3a shows a bump. This is caused by the elastic properties of the viscoelastic sample when the sample starts to flow. This transient effect is called overshoot-behavior.

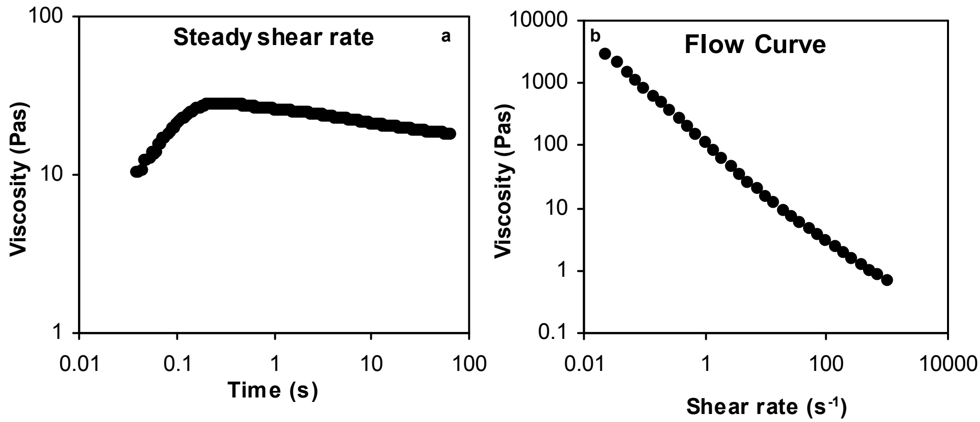


Figure 1.3a,b Example of (a) a steady shear-rate measurement and (b) of a flow-curve measurement.

In a flow-curve measurement, the stress and viscosity is determined as a function of strain rate (see Fig. 1.3b). The increase in shear rate can be compared to stirring at increasing speed. For many liquid (-like) materials, the rheological behavior during this measurement can be described by the Power Law:

$$\sigma = K \cdot \dot{\gamma}^n \tag{1.1}$$

where σ is the shear stress, $\dot{\gamma}$ the shear rate, K the consistency and n the flow index. Three types of behavior can be observed in this type of measurement. Samples with Newtonian behavior show a constant viscosity over the whole range of shear rates, resulting in a flow index n = 1 in the Power Law. Non-Newtonian samples adapt their structure when they flow, which leads to decreasing viscosities (shear-thinning behavior, $n < 1$) or increasing viscosities (shear-thickening behavior, $n > 1$) with increasing shear rates. Most semisolid food products show shear-thinning behavior, see Fig. 1.3b. When the sample is stirred faster, the relative force required to do so is lowered.

Many semisolid materials also show plastic behavior at the start of the flow curve; they do not start to flow immediately, but only above a certain minimum amount of stress. This threshold stress is called the yield stress and is included in adapted versions of the power law, for example that of Herschel-Bulkley or Casson:

Herschel-Bulkley: $\sigma = \sigma_Y + K \cdot \dot{\gamma}^n$ (1.2)

Casson: $\sqrt{\sigma} = \sqrt{\sigma_Y} + K \cdot \sqrt{\dot{\gamma}}$ (1.3) where σ_y is the yield stress. The yield stress of a material can be determined by rheological measurements with a specific set-up or by fitting of the flow curve with equations as shown above. This latter method was used in the studies of this thesis.

Literature on mayonnaises and custards

Shear rheological properties of mayonnaises and salad dressings have been investigated by many authors. Overviews are given by Rao (1992), Goshawk (1998) and Rao (2007). The latter also includes values measured for rheological properties. Most of the studies focused on the rheological properties in large deformation (Elliott & Ganz 1977; Pons *et al.* 1994; Juszczak *et al*. 2003) and determine values for the consistency, flow index, and yield stress of Eq. 1.1, 1.2 and 1.3. The overshoot and thixotropic behavior during steady shear stress measurements was studied and modeled by Tiu & Boger 1974, Kokini & Dickie (1981), Figoni &Shoemaker (1983), Campanella & Peleg (1987a), and Gallegos *et al*. (1992). Other publications include measurements at small deformation conditions next to large deformation measurements to determine both moduli in the LVER and the end of the LVER (Bistany & Kokini 1983; Muñoz & Sherman 1990; Peressini *et al.* 1998; Wendin & Hall 2001; Tabilo-Munizaga & Barbosa-Cánovas 2005). Traditionally, shear rheometry is performed by shearing the sample between two rotating surfaces, such as plate and plate, cone and plate or bob and cup. In recent studies, a vane geometry (comparable to a stirrer) is sometimes used to apply shear deformation. The advantage of this method is the limited damage to the sample in preparation of the measurement. For mayonnaises, vane geometries were applied by Štern *et al*. (2001, 2007) and Genovese & Rao (2005). Another group of publications (Ma & Barbosa-Cánovas 1995; de Cock & Vanhemelrijck 1995; Wendin *et al.* 1997; Pascual *et al*. 1999) investigate the effect of ingredients on the rheological properties of mayonnaises and salad dressings.

There are only a limited number of publications on shear rheometry of custards (Wischmann *et al*. 2002; Depypere *et al*. 2003; Verbeken *et al*. 2004; Tarrega & Costell 2006; and Ahmt *et al.* 2004), including related dairy products, such as egg-based custards (Longreé *et al.* 1966) and Spanish 'natillas' (Tarrega *et al*. 2004, 2005), almost all been published recently. More studies have been done on yoghurt (Rohm 1989; Rönnegård & Dejmek 1993; Skriver *et al*. 1993; De Lorenzi *et al*. 1995), which have been reviewed by Benezech & Maingonnat (1994).

Bulk rheometry in elongation

The measurement of rheological properties in elongation of liquid and semisolid materials is less standard than measurements in shear. To obtain elongational flow, the sample should be compressed or stretched (see Fig. 1.1). Several measurement techniques (Padmanabhan 1995) have been developed to measure rheological properties in elongation for semisolid and liquid products, such as the opposing jets technique (Steffe 1996), filament stretching (Rodd *et al.* 2005), and the tubeless siphon technique (Steffe 1996). These techniques are, however, not suitable to measure semisolid food products like custards and mayonnaises, because they are either too thick or too thin. Semisolid products with intermediate consistencies can be subjected to a four-roll mill system (Macosko 1994), but this technique is merely qualitative as it is difficult to control and determine forces and velocities. Another set of techniques suitable for custards and mayonnaises are empirical measurements in which a sample is forced to flow through an orifice, such as the Posthumus funnel (Osinga 1975; Hellinga *et al*. 1984), and forward- and backward extrusion (Steffe 1996). These techniques do not measure pure elongational behavior, however, because a combination of shear and elongational deformation is applied to the sample. It is often difficult to quantify the shear component within the results of these techniques.

A more useful, nonempirical technique is the squeezing flow technique (Chatraei *et al*. 1981; Steffe 1996; Campanella & Peleg 2002; Engmann *et al*. 2005), in which biaxial elongational deformation is achieved by compressing the product between two parallel plates. During compression, shear can be present next to elongational deformation, depending on the slip conditions at the plate-product interface. Pure elongational flow can be obtained by the lubricated squeezing flow technique (Chatraei *et al*. 1981), in which shear is minimized by maximizing the slip at the plate-product interface, which is achieved by applying lubricants or adjusting the surface of the plates. In this thesis, a special case of the squeezing flow technique is used; imperfect lubricated squeezing flow in a Teflon geometry (Hoffner *et al*. 1997; Suwonsichon & Peleg 1999). Imperfect refers to the wide, shallow container replacing the bottom plate (see Fig. 1.4). Lubrication is promoted by the Teflon surface of the container and upper plate (Suwonsichon & Peleg 1999). It is probably the only practically available, nonempirical technique usable to study biaxial elongation of semisolid food products such as custards and mayonnaises.

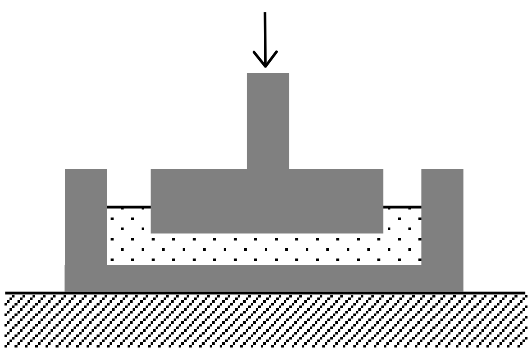


Figure 1.4 Set-up of the imperfect squeezing flow measurement in a Teflon geometry (Campanella & Peleg 2002).

An extra advantage of the technique is that the compression movement applied resembles the movement of tongue and palate during oral processing. The shallow container assures easy loading of the sample and maintains the sample without leakage during the measurement. Another advantage is the low level of damage to the sample during preparation for the measurement (Campanella & Peleg 2002). An important disadvantage of the imperfect technique is that the flow pattern is less well defined than in the ideal set-up, making the validity of the theoretically derived models questionable.

The imperfect squeezing flow measurement applied in this thesis, consists of three steps, see Fig. 1.5. In the first step, elongational flow is established by compression at constant velocity. The sample is compressed from the initial sample height to a final sample height of 1 mm. The force required to compress the sample is recorded. During compression, the strain rate increases, as it depends on the compression velocity as well as the sample height. When the compression occurs fully lubricated, the force versus sample height data can be calculated into a flow curve of elongational viscosity versus elongational strain rate, similar to that measured by shear rheometry. Extra information is obtained from the second and third step of the measurement. In the second step, the upper part of the geometry is halted at the final sample height for 2 min. This allows the measurement of stress relaxation of the sample after compression (Corradini *et al*. 2000a). Finally, a decompression step is applied, in which the upper part of the geometry is pulled up again. Next to elongational rheological properties, a number of empirical squeezing flow parameters are extracted as well from all three steps of the measurements (see Chapter 5 and 6).

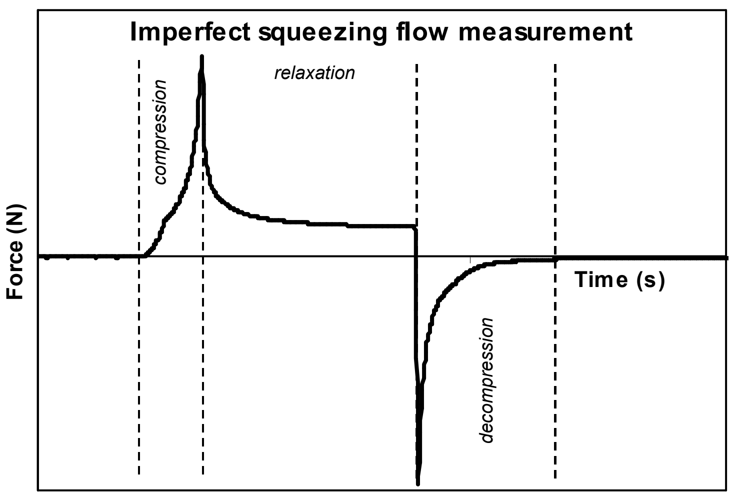


Figure1.5 Example of an imperfect squeezing flow measurement with subsequent steps.

Since the 80's, (imperfect) squeezing flow has been used to measure the properties of a range of semisolid foods, such as soft cheeses, peanut butter, tomato products and mustard (Casiraghi *et al.* 1985; Campanella & Peleg 1987b; Lorenzo *et al.* 1997). Measurements on mayonnaises have been reported amongst others by Hoffner *et al.* (1997) and Corradini *et al.* (2000a) and on yoghurts by Suwonsichon & Peleg (1999) and Janhøj *et al.* (2006). Some of the studies (Corradini *et al.* 2000a; Campanella & Peleg 2002) have shown that despite the use of Teflon, the requirement of full lubrication is not always fully met. This means that conditions during compression also involve some shear next to elongation.

Other instrumental measurements

Next to bulk rheometry, some other types of measurements are applied in Chapter 4 of this thesis. These involve measurements on other physicochemical properties, and/or on the changes of properties of the food during oral processing.

Measurements on other physicochemical properties

Measurements on other physicochemical properties in this thesis consist of friction measurements and fat droplet size measurements. These measurements cover other physicochemical phenomena and can therefore be a relevant addition to bulk rheometry. Friction measurements determine properties that have a background in tribology, also called thin-film rheology. These are properties associated with adhesion, wear, friction and lubrication. These properties describe the interaction of two surfaces that move relatively to each other, with or without a lubricating liquid or semisolid material in between. In this thesis, the frictional or lubricating properties of the material in between are measured. As these properties involve the interaction of only the thin film of sample with its surroundings, they are surface-related instead of bulk-related and cannot be determined directly by bulk rheometry. Friction of semisolids, expressed in a friction coefficient, is typically measured by applying a little amount sample between two surfaces that are sheared along each other. Characteristics of the measurement set-up are the types of materials used for both surfaces, the applied load, and the speed of shearing. Friction measurements on mayonnaises, custards and other semisolids have been reported by Giasson et al. (1997), Malone et al. (2003), de Wijk & Prinz (2005), de Hoog et al. (2006), and Dresselhuis (2008).

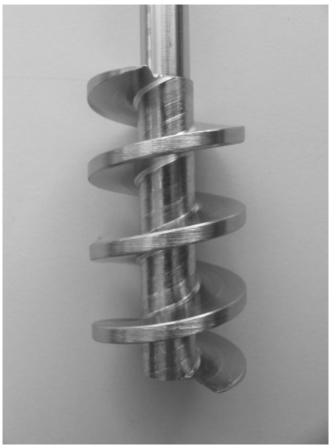
Information on the dispersed fat droplets can be obtained by fat droplet size measurements. These measurements are performed on a suspension of fat droplets extracted from the food matrix. This suspension is lead through a laser beam, which characterizes the size of the passing droplets. In this thesis, only the average droplet size, determined from the measured droplet size distribution, is used. Typical fat droplet sizes that have been measured for mayonnaises are approximately 5 μm (Kiosseoglou & Sherman 1983; Wendin et al. 1997).

Measurements on changes during oral processing
In the study of food perception, it is important to measure the changes in food properties during oral processing. These involve bulk rheological as well as other types of physicochemical properties. Determination of the changes in food properties can be performed in vivo or in vitro. In case of in vivo determinations, samples undergo oral processing, are then taken out of the mouth and are subjected to standard measurements as described above. An example of such a measurement is the determination of the turbidity of the rinse water (Prinz et al. 2006), which was recently developed in our project and applied in Chapter 4. It involves a turbidity measurement on the rinse water used to wash away the oral coating retained after swallowing the food product. This provides information on the composition (fat content, thickener phase) of the oral coating and the effect of saliva on the composition.

More information can be obtained from in vitro measurements, in which oral processing is mimicked in the measurement device, and the effect on food properties is simultaneously measured. It is very difficult though to design experimental conditions completely similar to oral processing, including the exact pattern of forces and velocities and the mixing with saliva. Conventional bulk rheological measurements are limited in mimicking oral processing. Small deformation rheometry determines only initial bulk properties, comparable to a food product directly after oral intake, without any oral processing. Large deformation rheometry can mimic the mechanical deformation that is orally applied to the food bolus, albeit in a simplistic form.

In the project that this thesis is part of, some novel instrumental techniques were designed that determine various types of physicochemical properties under more or less mouth-like conditions. These techniques are merely empirical, because the experimental set-up is often complicated and difficult to define exactly. One such technique is the friction measurement, adapted to mouth-like conditions (de Wijk & Prinz 2005), in which friction of a sample is determined between mouth-like surfaces (a rotating metal cylinder and a rubber band) and with the addition of saliva. Two other novel measurements can be used to study the general effect of saliva: the rate and extent of mixing of saliva into the product can be determined by in vitro mixing experiments in combination with image analysis (Prinz *et al.* 2007) and the overall effect of saliva on the composition of different products can be determined by measuring infra-red reflectance of *in vitro* samples with added saliva (de Wijk *et al.* 2006b). Results show that these latter measurements relate strongly to the fat content of the products and are representative of properties of the surface of the food bolus.

For the study of the effect of saliva on bulk rheological properties, a structure breakdown cell (SBC) was developed (Janssen *et al*. 2007). The cell consists of a helical rotating vane (see Fig. 1.6) and fitting cup, which can be attached to a standard rheometer. After addition of saliva to the sample in the cup, it is rapidly mixed through the bulk and subsequent changes in torque are followed in time. The torque is a measure of bulk viscosity. The measurements can be performed on the sample as such, with addition of water, or with addition of saliva (see Fig. 1.6). By comparing these three measurement curves, the effect of mechanical breakdown, of enzymatic breakdown of starch, and of dilution on viscosity can be separated from each other.



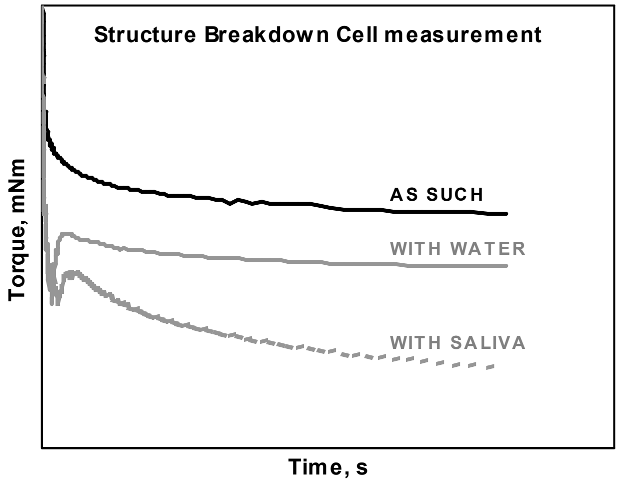


Figure 1.6 Helical vane of the structure breakdown cell and an example of curves measured by the SBC.

Relations between Texture Perception and Physicochemical Properties

Many studies (see Stanley & Taylor 1993; Guinard & Mazzucchelli 1996; van Vliet 2002) have been performed in the last decades to unravel the relations between orally perceived texture attributes and physicochemical properties for semisolid foods and related products. The focus of many of these studies is on perceived thickness and the effective oral shear rate. Physicochemical properties that were measured consisted primarily of bulk rheological properties in shear.

The relations between texture attributes and physicochemical properties were obtained by modeling, univariate correlations or multivariate predictions. Modeling is the most sophisticated and difficult way to relate texture perception to physicochemical properties; models are established in which the assumed physicochemical properties underlying texture perception are calculated from initial food properties by simulating oral processing conditions during texture perception. This requires knowledge on the mechanisms underlying texture perception, on oral processing conditions and on the way the physicochemical properties of the food change during oral processing and leads often to complicated equations. A more empiric approach is to find relations within a data set of texture attributes and specific physicochemical properties or parameters by univariate correlation or multivariate prediction. In univariate correlation, one-on-one relations are obtained, while in multivariate predictions, texture attributes can be related to a combination of physicochemical properties. Multivariate methods are only recently available for application, but they are very promising as texture attributes often originate from a combination of properties.

Most studies focus on the physicochemical properties that are related to texture attributes and neglect the exact nature of their relations. In univariate correlations and multivariate predictions, the nature of the relation is considered linear; the attribute is (inversely) proportional to the physicochemical property. For a large number of human perceptions however, the relations between sensory attributes and a physical or chemical stimulus have been found to be nonlinear (Coren *et al*. 1999). These relations are often semilogarithmic (Fechner 1860) or follow a powerlaw (Stevens 1975):

Semilogarithmic: $$S = k_1 \cdot \log(I/I_0) \quad (1.4)$$

Powerlaw: $$S = k_2 \cdot I^w \quad (1.5)$$

where S is the (sensory) sensation intensity, I is the physical stimulus intensity, k_1 and k_2 are proportionality constants, I_0 is the absolute threshold stimulus intensity, and w is the powerlaw constant.

Despite all the research, the exact mechanisms underlying attributes such as creaminess, fattiness, and melting are still not fully understood. Even thickness, a relatively straightforward and viscosity-related attribute, cannot be identified completely by one predicting rheological property or parameter. Oral perception of most texture attributes seems to proceed in a very complex way; it is the result of a combination of properties, which have different physicochemical backgrounds.

Modeling

Modeling of oral texture perception has been done primarily for the attribute thickness. One such model is proposed by Kokini *et al*. (1977) and is based on the assumption that thickness is perceived as the shear stress on the tongue at the moment of perception. By applying fluid mechanics, two physical-physiological models are established that predict orally perceived thickness from rheological and physiological parameters reflecting oral conditions. The models simulate the movements of the tongue and palate and calculate the effect of this deformation on the shear stress on the tongue of the food bolus. One model is relatively simple and simulates only a lateral movement of tongue and palate (shear flow):

$$Thickness \cong K \cdot \left(\dot{\gamma}_{tongue}\right)^{n} \tag{1.6}$$

The other model is more complicated and simulates the combination of a lateral movement of the tongue and a compression movement of the tongue towards the palate (shear and elongational flow):

$$Thickness \cong K \cdot \frac{V^{n}}{h(t)^{n}} \tag{1.7}$$

with

$$h(t) = \left[\frac{1}{h_0^{(n+1)/n}} + \left(\frac{F_0}{R^{n+3}} \cdot \frac{n+3}{2\pi K} \right)^{1/n} \cdot \frac{n+1}{2n+1} t \right]^{-n/(n+1)}$$

where Thickness is the orally perceived thickness. Physiological parameters consist of $\dot{\gamma}$, the (constant) shear rate exerted by the tongue; V, the average tongue velocity lateral to the palate; h(t), the height of the fluid layer between tongue and palate as a function of time; h_0, the initial height of fluid layer on the tongue; F_0, the compression force or load exerted by tongue towards the palate; R, the effective radius of tongue; and t, the time required for assessment of thickness. Consistency K and flow index n are the power-law constants obtained from flow-curve measurements describing the shear rheological behavior of the food product.

Limitations of these models are that they only describe the mechanical deformation and breakdown during oral processing. Other factors, such as the effect of saliva are neglected. In addition, the model calculates only behavior of liquids, hence, yield stress and viscoelasticity are not included. Furthermore, the shape of the mouth and the oral movements are simplified to two parallel hard plates that slide along each other and approach towards each other. In reality this movement more like a wedge, which moves from the front of the mouth to the back to induce swallowing (Tasko *et al.* 2002).

Another model on thickness perception has been established by Chen (1993), in which a wedge-like oral movement is modeled. Dickie & Kokini (1983) proposed a model in which thickness is assumed to originate from the overshoot phenomenon. Models established for other oral texture attributes of semisolid foods include smoothness, perceived as the inverse of the friction force on the tongue and slipperiness, perceived as a combination of viscous and friction forces on the tongue (Kokini *et al*. 1977). Another model by Steffe *et al.* (2003) described slipperiness in terms of a lubricity index, based on the effect of temperature and dilution.

Univariate correlation and multivariate prediction

Univariate correlations of orally perceived texture attributes with physicochemical properties that have been published for semisolids foods often involve conventional, well-known rheological properties such as the flow index from the flow-curve measurements and dynamic moduli in the LVER from dynamic measurements. Table 1.2 and 1.3 give an overview of the physicochemical properties related to creaminess and other oral texture attributes of semisolids as reported in literature. Relations with properties obtained from empirical measurements such as the Texture Profile Analysis (TPA), for example on Baker's custard (Kampp 1995) are not included.

Table 1.2 Univariate correlations in literature for creaminess. Fc, ssr, and ds stand for flow-curve, steady shear-rate, and dynamic sweep measurements.

Physicochemical property (measurement)	Type of products	
Viscosity at 50 s-1 (fc)	O/W emulsions with butterfat	Akhtar et al. 2005
Friction	custards	de Wijk & Prinz 2006
Consistency K (fc) Flow index n (fc)	custards	Ahmt et al. 2004
G", loss modulus in LVER (ds)	ice creams	Wildmoser et al. 2004
Air bubble size Total volume of included air G', G" moduli in LVER (ds) Viscosity at 1 s-1 (fc) TA penetration	model chocolate mousses	Kilcast & Clegg 2002 Clegg et al. 2003
Particle size and concentration	xanthan gels with hard particles	Kilcast & Clegg 2002
Fat droplet size	model cream emulsions	Kilcast & Clegg 2002
Optimum with viscosity at 50 s-1 (fc) Optimum with flow index n (fc)	model soups	Daget & Joerg 1991
Optimum with viscosity at 50 s-1 (fc) Optimum with flow index n (fc)	model caramel dessert creams	Daget et al. 1987
Fat droplet size, viscosity	creams and thickened milk	Richardson et al. 1993
Viscosity	milks	Shoemaker et al. 1992
Consistency K (fc) Viscosity at all shear rates (fc)	mayonnaises	Tunaley et al. 1985
Viscosity below 50 cps (fc)	soups	Wood 1974

Regarding the attribute thickness there has been a large debate in literature on the exact shear rate for viscosity measurements relating to orally perceived thickness (see Table 1.3). As the viscosity of most semisolid foods change with shear rate, establishing this shear rate value can give an indication on the shear rate that can represent deformation during oral processing.

In the past few years, multivariate analysis has been used more and more to express the complex relations of oral texture attributes with combinations of physicochemical properties (de Cock & Vanhemelrijck 1995; Tepper & Kuang 1996; Štern *et al.* 2001; Tarea *et al.* 2004; Ahmt *et al.* 2004). Janhøj *et al.* 2006 predicted creaminess, thickness and some other oral texture attributes of yoghurts from a combination of parameters from the Posthumus funnel, imperfect squeezing flow measurements and shear rheometry.

Table 1.3 Univariate correlations in literature for some other oral texture attributes. Fc, ssr, and ds stand for flow-curve, steady shear-rate, and dynamic sweep measurements.

Attribute	Physicochemical property	Type of products	
Thickness	G' storage modulus in LVER (ds) yield stress (fc)	model mayonnaises	Štern *et al.* 2007
	critical stress (ds)	salad dressings	Tornberg *et al.* 2004
	G" loss modulus in LVER (ds) critical stress (ds)	low-fat model mayonnaises	Wendin *et al.* 1997
	consistency K (fc) viscosity at all shear rates (fc)	mayonnaises	Tunaley *et al.* 1985
	consistency K (fc) flow index n (fc)	custards	Ahmt *et al.* 2004
	complex viscosity at 50 rad/s (ds)	thickened milk products	Richardson *et al.* 1989
	Brookfield viscosity at 100 rpm (ssr)	range of semisolids	Szczesniak *et al.* 1963
	viscosity at specific shear rates:		
	50 s^{-1}	model soups	Daget & Joerg 1991
		model caramel dessert creams	Daget *et al.* 1987
	50 s^{-1}	cream soups and sauces	Wood 1968
	10 s^{-1} and 50 s^{-1}	range of semisolid foods and related products	Cutler *et al.* 1983
	30 s^{-1}	ice creams	Aime *et al.* 2001
	10 s^{-1} and 300 s^{-1}	milk beverages	Yanes *et al.* 2002
Fattiness / perceived fat content	YS (fc) Jor (creep recovery)	model salad dressings	Wendin & Hall 2001
	G' LVER (ds) YS (fc) max viscosity (fc)	low-fat model mayo	Wendin *et al.* 1997
	viscosity (fc)	thickened milk products	Mela 1988
	viscosity (fc)	O/W emulsions	Mela *et al.* 1994
	friction (fc)	O/W emulsions	Malone *et al.* 2003
Mouthcoating	viscosity by Posthumus funnel	stirred yoghurts	Martin *et al.* 1999
	viscosity at 30 s^{-1} (fc) consististency K (fc)	ice creams	Aime *et al.* 2001
Slimy	flow index n (fc)	soups	Wood 1974
	complex viscosity at 50 rad/s (ds)	thickened milk products	Richardson *et al.* 1989

Outline of this Thesis

The central theme of this thesis is to identify relations between orally perceived texture attributes of semisolid foods and physicochemical properties, as well as to find instrumental measurements other than bulk rheometry in shear that can measure these properties. All studies include mayonnaises, some also custards. In **Chapter 2**, the oral perception of thickness is modeled for mayonnaises and custards. To this end, the applicability of the physical-physiological model of Kokini *et al*. (1977) is tested and attempts are made for improvements.

In **Chapter 3 and 4**, relations of oral texture attributes with parameters describing physicochemical properties are identified for mayonnaises by univariate correlation (Chapter 3) and multivariate prediction (Chapter 3, Chapter 4). Chapter 3 establishes these relations with bulk rheological properties in shear for a set of commercial mayonnaises with large variations in fat content. In Chapter 4, the general applicability of the results of Chapter 3 is tested for a larger and broader set of model and commercial mayonnaises and by multivariate prediction. In addition, other physicochemical properties from novel instrumental measurements are included in the predictions to assess whether these measurements, reflecting nonrheological properties and/or properties at mouth-like conditions, can improve the predictions from bulk shear rheological properties alone.

Chapter 5 and 6 investigate the relevance of bulk rheological properties in elongation, obtained by the imperfect squeezing flow technique, in relation to the oral texture perception of mayonnaises and custards. In Chapter 5 the measurement technique is explored for the measurement of elongational behavior of mayonnaises and custards. In Chapter 6, the measurement technique is applied to characterize a set of commercial mayonnaises and a set of commercial custards and the measured properties related to texture attributes in a univariate and multivariate way. Next to this, the yield stress and flow index obtained from the imperfect squeezing flow measurement is compared to those obtained from rheological measurements in shear.

In **Chapter 7**, the main findings of the previous chapters are combined and discussed.

2

MODELING OF THICKNESS FOR SEMISOLID FOODS

Abstract

We investigated models that predict orally perceived thickness from calculated shear stress on the tongue for mayonnaises and custards. To this end, the applicability of the physical-physiological models of Kokini *et al*. (1977), describing the mechanical breakdown in the mouth, were tested and attempts were made for improvements. Within a limited range of shear stresses (mayonnaises < 150 Pa; custards < 30 Pa), there was a linear relationship between shear stress and thickness, in accordance with the work of Kokini *et al*. (1977). Beyond this range, the linear relationship breaks down and the thickness levels off with shear stress for both mayonnaises and custards. The relationship over the entire range of shear stresses used in this paper can be satisfactorily described by a semilogarithmic (Fechner's) relation. For both types of products, the quality of the thickness prediction by the decreasing-height model and the constant-height model of Kokini *et al*. (1977) is similar. For most mayonnaises, the contribution of the lateral movement of the tongue to the shear stress in the decreasing-height model of Kokini *et al.* (1977) is orders of magnitude larger than the contribution of the squeezing or compression movement of the tongue towards the palate. This difference in magnitude is affected by the low value measured for the compression force, and by the high values for material consistency K. The values for K are high because yield-stress behavior has been neglected when the flow curves were analyzed. For custards, the models of Kokini *et al.* (1977) are found to be less adequate. It is proposed that this is because the models ignore interactions with saliva. Several routes to improve the modeling by incorporating viscoelastic behavior were unsuccessful. Elongational stress and yield stress were neglected in all tested models.

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*M.E.J. Terpstra, A.M. Janssen, J.F. Prinz, R.A. de Wijk, H. Weenen and E. van der Linden, Journal of Texture Studies **2005**, 36, 213-233*

Introduction

Although it seems obvious to predict orally perceived thickness of semisolid foods from rheological data, in practice it turns out to be difficult to appoint one single representative rheological parameter. While some researchers (Daget *et al.* 1987; Richardson *et al*. 1989; Wendin *et al*. 1997; Štern *et al*. 2001) use an empiric approach to correlate perceived thickness to rheological parameters, others try to find the mechanisms and conditions of deformation representative for oral processing. These latter studies focus mainly on the effective shear rate representative for oral deformation (Wood 1968; Shama & Sherman 1973a; Cutler *et al*. 1983; Houska *et al*. 1998). Considerable speculation still exists on the actual effective oral shear rate, especially in the case of weak gels (Stanley & Taylor 1993). Another approach in predicting perceived thickness from rheology is to establish models that simulate mechanisms and deformation conditions in the mouth during human thickness assessment. One such model is proposed by Kokini *et al*. (1977) and is based on the assumption that thickness is perceived as the shear stress on the tongue during assessment. By applying fluid mechanics, this assumption has led to two models that predict orally perceived thickness from rheological and physiological parameters reflecting oral conditions. An improved version of the model, including transient effects has been published by Dickie & Kokini (1983).

All studies mentioned above only consider mechanical deformation during oral processing. In reality however, food is also subjected to thermal equilibration and dilution with saliva during mastication (Engelen *et al.* 2003; Engelen *et al.* 2005). Starch-containing food products will also undergo enzymatic breakdown due to mixing with α-amylase from saliva (de Wijk *et al.* 2004; Janssen *et al.* 2007).

In this study we investigate the relationship between orally perceived thickness and calculated shear stress on the tongue for two types of viscous semisolid food products. Previous studies, using the same panel, attributes and types of products as the current study show that thickness is perceived initially after intake of the food product (de Wijk *et al.* 2003a,b). At the same time, it appears that some oral processing is required to assess thickness (de Wijk *et al.* 2003a), as thickness correlates mainly to rheological properties measured under deformation, and to a much lower extent to properties measured at rest (Janssen *et al.* 2007; see Chapter 3 and 4). The aim of the study is to determine the applicability of the models of Kokini *et al*. (1977) to describe the mechanical breakdown in the mouth of two types of food products, i.e., mayonnaises and custards. Custard is a typical Dutch dairy-dessert product, consisting mainly of milk and starch. Both types of products can be considered as weak gels, showing yield stress, thixotropy and strong shear-thinning behavior (see Chapter 3). Although this study focuses on only two types of products, there is a large variation in texture properties within each product type due to variations in ingredients and processing conditions.

Background

Kokini *et al*. (1977) presented a model that relates thickness as perceived in the mouth to rheological and physiological variables. This model was originally developed to predict thickness of fluids as perceived between fingers (DeMartine & Cussler 1975) and represents only mechanical breakdown during oral processing. The model assumes that thickness varies with the shear stress felt on the tongue during assessment. Kokini *et al*. (1977) reported this relationship between perceived thickness and the shear stress according to

$$Thickness \cong a \cdot \sigma^b \tag{2.1}$$

where Thickness is the orally perceived thickness, σ is the shear stress felt on the tongue during assessment, a and b are constants. The exponent b varies between 0.89 (Kokini *et al*. 1977) and 0.97 (Elejalde & Kokini 1992), from which both authors conclude that the relation between perceived thickness and shear stress can be considered linear. The shear stress felt on the tongue is a function of flow within the mouth. This flow is assumed to result from a combination of a lateral movement of the tongue (shear flow), and a squeezing or compression movement of the tongue towards the palate. Fluid mechanical calculations (DeMartine & Cussler 1975) quantify the shear stress, and according to Eq. 2.1, with b = 1, the following expression for thickness was found

$$Thickness \cong K \cdot \frac{V^n}{h(t)^n} \tag{2.2}$$

with

$$h(t) = \left[\underbrace{\frac{1}{h_0^{(n+1)/n}}}_{\text{lateral movement}} + \underbrace{\left(\frac{F_0}{R^{n+3}} \cdot \frac{n+3}{2\pi K} \right)^{1/n} \cdot \frac{n+1}{2n+1} t}_{\text{compression movement}} \right]^{-n/(n+1)} \tag{2.3}$$

where V is the average tongue velocity lateral to the palate during the assessment of thickness, h(t) is the height of the fluid layer between tongue and palate as a function of time, h_0 is the initial height of fluid layer on the tongue, F_0 is the compression force or load exerted by tongue towards the palate, R is the effective radius of tongue, t is the time required for assessment of thickness. Consistency K and flow index n are the power-law constants describing the rheological behavior of the food product. Values of K and n for a particular fluid can be determined from flow-curve measurements. The first term of h(t) denotes the contribution due to the lateral movement of the tongue, while the second term describes the shear stress due to the compression movement of the tongue. As a whole, Eq. 2.2 and 2.3 calculate the shear stress arising at time t, at the end of the thickness assessment. In the derivation it is assumed that viscosity and shear rate follow power-law behavior, and that F_0 is constant, i.e., that the compression movement of the tongue takes place at constant force, and not at constant velocity. We will refer to the model in Eq. 2.2 as the 'decreasing-height model'. The decreasing-height model can be simplified by assuming that the load exerted by the tongue, F_0, is zero. In this special case the oral movement only results from a lateral movement of the tongue, such that the height of the fluid layer remains constant during the assessment of thickness. In this special case, Eq. 2.2 simplifies to:

$$Thickness \cong K \cdot \left(\dot{\gamma}_{tongue}\right)^{n} \qquad (2.4)$$

where $\dot{\gamma}$ is the (constant) shear rate exerted by the tongue, replacing the ratio of velocity over height. We will further refer to the model in Eq. 2.4 as the 'constant-height model'.

Viscoelastic behavior

Many semisolid (food) materials have an internal structure of some sort, which is shown experimentally as yield stress and/or viscoelasticity. For these products, simple power-law behavior is not sufficient to fully describe their behavior, which includes e.g., transient effects. When viscoelastic materials are suddenly deformed, the initial reaction has an elastic nature, followed by viscous changes. This phenomenon can be seen in curves of steady shear-rate measurements, in which shear stress will first increase rapidly, and then decrease gradually to an equilibrium. This is also known as overshoot behavior. The time span of the initial, elastic reaction is governed by the relaxation time of the material. Dickie & Kokini (1983) stated that because relaxation times of food products can be relatively large and are comparable to times associated with thickness assessment, it is likely that transient viscoelastic effects play a role in perception of thickness. Because power-law behavior is not valid in this case, an improved thickness model has been published by Dickie & Kokini (1983) to account for this effect. In this improved model, perceived thickness is assumed to be related to the maximum overshoot stress, σ_{max}, felt in the mouth at the onset of processing, instead of the equilibrium stress in the previous models, according to:

$$Thickness \cong \sigma_{\max} \qquad (2.5)$$

This peak shear stress can be calculated from a fluid-mechanics model incorporating the overshoot effect described by the Bird-Leider equation (Leider & Bird 1974; Dickie & Kokini 1983).

Another result of internal structure is the presence, next to a shear stress, of a normal-stress component when these materials undergo shear deformation. This normal stress, which is perpendicular to the direction of shear, is defined by the first normal-stress difference N_1 (Steffe 1996). We hypothesize that normal stresses, generated by oral movements of the semisolid food, are sensed by the mouth and contribute to the perception of thickness, together with the shear stress sensed. For the perception of thickness, one may combine shear stress and first normal-stress difference using either a serial (Eq. 2.6) or harmonic approximation (Eq. 2.7), defined by:

$$Thickness \cong \sigma + N_1 \tag{2.6}$$

and

$$Thickness \cong \frac{\sigma \cdot N_1}{(\sigma + N_1)} \tag{2.7}$$

Another way to account for normal stress felt in the mouth is to incorporate it in the force exerted by the tongue to decrease the fluid layer between tongue and palate. This implies that the actual force applied to the fluid, as used in Eq. 2.3, is equal to the force applied by the tongue muscle, F_0, minus the force representing the normal-stress difference N_1.

Nonlinear psychophysical relations

So far, the exact relation between thickness and the shear stress calculated by the models has not been considered in detail. For a large number of human perceptions, the relations between sensory attributes and a physical or chemical stimulus have been found to be nonlinear (Coren *et al*. 1999). One of the first nonlinear relationships found is the semilogarithmic relation by Fechner (1860), according to:

$$S = k_1 \cdot \log(I/I_0) \tag{2.8}$$

where S is the (sensory) sensation intensity, I is the physical stimulus intensity, k_1 is the Fechner proportionality constant and I_0 is the absolute threshold stimulus intensity.

Stevens (1975) introduced a power relation to describe perception, given by:

$$S = k_2 \cdot I^w \tag{2.9}$$

where k_2 and w are constants. The shape of the curve from Eq. 2.9 depends largely on the value of w, but for $0 < w < 1$ an increasing curve with decreasing slope is observed for Stevens's as well as Fechner's relationship. For the perception of thickness, a Stevens's power relation has been hypothesized (DeMartine & Cussler 1975; Kokini *et al.* 1977; Elejalde & Kokini 1992), but the authors conclude from their results that the relation between thickness and the physical stimulus can be considered linear.

Materials and Methods

Materials

Thirty-eight model mayonnaises were produced by TNO Nutrition and Food Research Institute (Zeist, The Netherlands). Five of the mayonnaises were full-fat-mayonnaises, containing 80% soy oil and no thickeners. The other samples contained 5-40% soy oil and were in most cases thickened by a combination of starch, xanthan and/or guar gum. Variations between the samples included amount and type of starch, other thickeners and emulsifiers, amount of oil and processing conditions.

Twenty-three model custards were produced by NIZO food research (Ede, The Netherlands). Variations between the samples included type and amount of starch and amount of milk fat (0-15%).

Sensory methods

Sensory scores for perceived thickness have been assessed by a sensory panel trained in quantitative descriptive analysis (QDA; Stone & Sidel 1985). Thickness was scored along with other odor, flavor, mouthfeel, and afterfeel attributes on a 0-100 points unstructured scale. The panel consisted of nine subjects, aged between 22 and 49 years, who were not overweight. The subjects had previously been screened for olfactory and taste disorders and had received extensive training in the use of sensory odor, flavor, texture, and afterfeel attributes for mayonnaises and custards. The subjects were paid for their participation. Panel testing took place at the sensory facilities of TNO Nutrition and Food Research Institute. Subjects were seated in sensory booths with appropriate ventilation and lighting. Each set of products was measured in three sessions preceded by an extra training session. Every session took 2 h, in which all samples were presented once in a randomized order. Over three sessions, held at the same day and time at 3 consecutive weeks, subjects were presented with three replicates of each sample. During presentation of a sample, subjects rated odor, flavor, mouthfeel, and afterfeel attributes using a mouse and a 100-point line scale presented on a computer screen. All attributes had been previously generated using a QDA protocol. A more detailed description of the procedures has been given by de Wijk *et*

al. (2003b). For the present study, only scores for thickness have been used. Thickness has been defined by the panel as the thickness of the food in the mouth after the food is compressed via up-and-down motions of the tongue against the palate; for less viscous products information is also obtained from the rate of spreading of the product through the mouth. Thickness is one of the first attributes rated when a spoonful of product is delivered in the mouth.
Thickness scores have been averaged over all panel members. Scores of four sets of model mayonnaises were mixed to create a large dataset for modeling. Similarly, results for custards were obtained by combining two datasets. Graphs with sensory thickness versus model shear stress (like Fig. 2.1) for individual datasets per type of product completely overlapped each other, confirming that mixing of the sets of sensory scores is allowed for both types of products.

Rheological methods

Flow curves and steady shear-rate measurements of all the samples have been determined with a Paar Physica MCR 300 rheometer, equipped with a 40 mm plate/plate system. To prevent slip, grooved plates and plates with sandpaper (kor 80) stuck on it have been used for measurements on custards and mayonnaises, respectively. Flow-curve measurements were performed from 0.01 to 1000 s^{-1} (36 measuring points, 15 s per point). Steady shear-rate measurements were performed at 10 s^{-1} for 1 min (200 measuring points, taken logarithmically in time). Samples were carefully loaded on the bottom plate with a plastic spoon to minimize structure breakdown. After lowering the upper plate to a gap width of 1.050 mm, excess of sample was removed with a plastic spatula. A final gap distance of 1 mm was applied, and samples were allowed to relax and acclimatize before measurements were started. Waiting time and applied temperature depended on the type of products; 5 min, 20°C and 10 min, 22°C for custards and mayonnaises, respectively. A cover was placed over the measuring system to prevent dehydration of the sample. Measurements were done in duplicate or triplicate.

Flow curves of both types of products could be described by the powerlaw equation. From the intercept and slope of the logarithmic plot of the curve between 0.1 and100 s^{-1} consistency K and flow index n were calculated, respectively. Yield-stress values of the products were quantified by fitting stress versus shear-rate data from flow-curve measurements by the Herschel-Bulkley model (Steffe, 1996). This was done for all custards and for a representative group of mayonnaises. For one of the mayonnaise sets also first normal-stress differences have been recorded, and values obtained at 30 s^{-1} were used for calculations.

Steady shear-rate measurements were used to determine overshoot peak stresses. The maximum stresses were normalized with the equilibrium stress, as described by Dickie &

Kokini (1983). It was difficult to establish the equilibrium value, because both types of products behave thixotropically, therefore the stress after 60 s shearing has been taken as the equilibrium stress.

Physiological methods

The required physiological parameters (see Table 2.1) are assumed to be the same for both types of products. We expect oral processing of these samples to be similar, as texture characteristics of both types of products are in the same range. Compression force exerted by the tongue has been estimated during eating of custard by a combination of a glass ball and pressure-sensitive paper, both wrapped in a balloon (Prinz, unpublished results). The average color intensity of the track at the pressure-sensitive paper was compared with the intensities obtained by moving three balls around under different weights. Tongue radius has been estimated by measuring bite marks after biting a piece of paper. The initial height of the fluid layer and the time required for thickness assessment has been estimated based on bite size and panel observations.

Table 2.1 Values estimated for physiological model parameters.

Effective radius of tongue, R	2 cm
Initial height of fluid layer, h_0	0.5 cm
Compression force exerted by tongue, F_0	0.25 N
Time required for assessment, t	0.3 s

Results and Discussion

Rheological, physiological and sensory values

Table 2.2 shows the range of values for the rheological parameters K and n and for the averaged scores of orally perceived thickness assessed for mayonnaises and custards. Physiological parameters estimated are given in Table 2.1. The value for the compression force exerted by the tongue was found to be 0.25 N, which is somewhat lower than values reported in literature (Kokini *et al*. 1977; Dickie & Kokini 1983; Miller & Watkin 1996), which range between 0.3 and 4 N. The difference between our and their results might be caused by the use of other methods to determine the force and/or the measurement of forces during normal mastication of semisolids instead of during swallowing. Another reason may be the large variation in applied force between human subjects, as reported by Miller & Watkin (1996). In their study, values of peak forces range between 0.06 and 3.8 N for different subjects. Other physiological parameters, like R, h_0 and t have been estimated as well (see Table 2.1). The assessment time t was estimated to be 0.3 s, because a mastication cycle typically takes 1 s, which is divided equally over three phases; tongue up, pause and tongue down. For the average lateral velocity of the tongue (for the decreasing-height model) and shear rate (for the constant-height model), we did not estimate a specific value, but calculations on the models were performed for several shear rates in the range of 0.1-200 s^{-1}. Tongue velocity was calculated from the shear rate taken by $\dot{\gamma} = V/h_0$.

Table 2.2 Range of the rheological values and sensory scores determined for mayonnaises and custards.

	Mayonnaises	Custards
Consistency, K (power law)	17.9 – 198.2 Pas^n	9.7 – 46.4 Pas^n
Flow index, n (power law)	0.04 – 0.38	0.18 – 0.37
Orally perceived thickness	14 – 72	15 – 69

The sensitivity of the decreasing-height model for R, h_0, F_0 and t has been tested by analysis of the powers of the physiological parameters in Eq. 2.2, using the n-values found in this study. Analysis shows that the model is most sensitive to changes in tongue radius, R, and also sensitive to changes in h_0 and F_0 (results not shown). The time required for assessment, t, has a minor influence on the stress calculated by the decreasing-height model. The above still holds when a realistic variation for each specific physiological parameter is considered. The effect of K on the outcome of the decreasing-height model is larger than of any of the physiological parameters, as was also shown by Kokini *et al.* (1977).

Application of models

Thickness has been plotted against the shear stress as calculated by the decreasing-height model (Eq. 2.2) and the constant-height model (Eq. 2.4). See Fig. 2.1a,b for mayonnaises and custards, respectively. Calculations were performed for several V and $\dot{\gamma}$. The values of $\dot{\gamma}$ that resulted in the best fit, i.e., with the highest linear correlation coefficient, have been used for Fig. 2.1. For mayonnaises, this shear rate was 30 s^{-1}, for custards 1 s^{-1}. The value of 30 s^{-1} for mayonnaises lies within the range of shear rates of 10-50 s^{-1} reported in literature for various semisolids (Wood 1968; Shama & Sherman 1973a; Cutler *et al*. 1983; Dickie & Kokini 1983). In this regard, the value found for custards seems unrealistically low. Varying $\dot{\gamma}$ and V within reasonable limits affects the quality of the fit, but does not affect the relative location of the data points.

Both graphs clearly show a relationship between calculated shear stress on the tongue and the orally perceived thickness. In this context, we want to address three issues: (a). The results seem to indicate that at higher shear stresses the perceived thickness levels off, (b) results for the decreasing-height and constant-height model are similar and (c) the results for custards are somewhat less good and imply a low value for the oral shear rate.

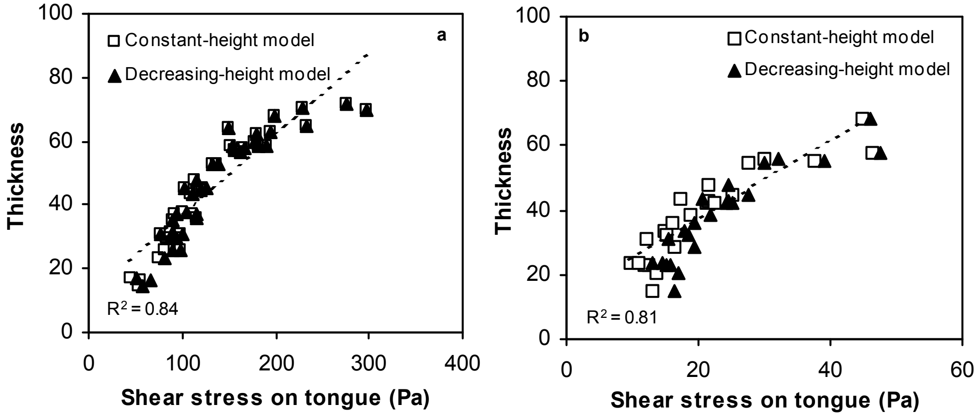

Figure 2.1a,b Orally perceived thickness versus calculated shear stress using the decreasing-height and constant-height model for (a) mayonnaises (at 30 s^{-1}) and (b) custards (at 1 s^{-1}). The dotted line shows the linear fit of the constant-height model data.

Leveling off at higher shear stresses

Kokini *et al*. (1977) and Elejalde & Kokini (1992) concluded that the relation between perceived thickness and calculated shear stress is linear. Our data indicate that perceived thickness levels off at higher shear stresses (>150 Pa for mayonnaises and >30 Pa for custard). This is especially pronounced for mayonnaises. This leveling-off effect does not depend on the choice of the physiological parameters, when these are varied within reasonable limits. A possible explanation for the effect might be the use of linear scaling between 0 and 100 in this paper. This can cause individual panel members to 'run out of scale' when assessing thick samples, leading to leveling off of our data points. However, this mechanism seems not to occur in our experiment, because the leveling off was still present after omitting panel members who gave scores close to 100. We therefore conclude from our data that at higher shear stresses the linear relationship with thickness perception breaks down. A reason for the absence of the leveling-off effect in the results of Kokini *et al*. (1977) and Elejalde & Kokini (1992) might be that their data points range only to 200 Pa, which makes it difficult to see the effect at higher shear stresses. The nonlinear behavior between perceived thickness and physical stimulus has been studied further by fitting to Fechner's and Stevens's relation (Eq. 2.8 and 2.9). For our data, the best fit is obtained with the semilogarithmic Fechner's relation (see Fig. 2.2a,b for mayonnaises and custards, respectively). The squared correlation coefficient of this fit is 0.92, versus 0.84 for the linear relationship for mayonnaises. For custards these values are 0.87 and 0.81, respectively. Correlation coefficients obtained with Stevens's power behavior are lower for both types of products, because the leveling off at higher shear stresses is not well fitted.

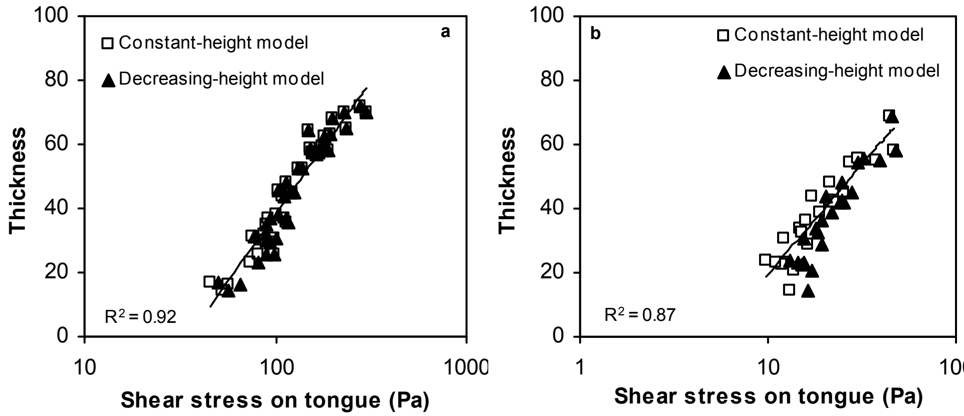

Figure 2.2a,b Decreasing-height and constant-height model fitted with the semilogarithmic Fechner's relation for (a) mayonnaises (at 30 s^{-1}) and (b) custards (at 1 s^{-1}). The solid line shows the fit of the constant-height model data.

Previous authors (Wood 1968; Shama & Sherman 1973b; Christensen 1979; Cutler *et al.* 1983; Houska *et al.* 1998) also reported a nonlinear relation between thickness and viscosity, in contrast to Kokini *et al.* (1977) and Elejalde & Kokini (1992). Although most of these researchers find a Stevens's power relationship to describe the nonlinearity between perceived thickness and physical stimulus (Wood 1968; Christensen 1979; Cutler *et al.* 1983), our data indicate a semilogarithmic (Fechner's) relationship. This type of relationship was also found by Houska *et al.* (1998) for fluid Newtonian food samples. One possible explanation for the different relationships reported could be the difference in sensory methods used; researchers finding Stevens's or linear relationships use magnitude estimation, while our sensory scores and that of Houska *et al.* (1998) were scored on a 0–100 scale without any reference sample, and lead to a Fechner's relationship. This is further supported by the work of Shama & Sherman (1973b) in which both sensory methods were used. Stevens's power relationship between sensory scores and physical stimulus has also been associated before with the use of magnitude estimation for obtaining sensory scores (Stevens 1975).

Another reason for the observed leveling off may be a change in oral-processing conditions upon mastication of thicker samples. For example, Shama & Sherman (1973b) and Houska *et al.* (1998) showed that oral shear rate decreases with increasing viscosity. One implication of this observation is that the choice of physiological parameters in a model is only valid within a limited range of rheological product properties, and that when these properties change one would have to adjust the physiological parameters. Another more severe implication is that the validity of the model itself has a limited range, because it specifically assumes the shear stress to be the mechanism to perceive thickness. Shama & Sherman (1973a), however, discriminated two regimes; at low viscosities, thickness is perceived as the shear rate at a specific shear stress, while at high viscosities, thickness is perceived as the shear stress at a specific shear rate. The same is suggested by the panel members in their definition of thickness; while in general thickness is assessed as the result of the compression of the product by up-and-down motions of the tongue against palate, for less viscous products information is also obtained from the rate of spreading of the product. We note that in this paper the oral physiological parameters such as the tongue velocity and shear rate have been kept constant for all samples, because it was assumed that oral processing conditions are the same within one type of products. This is probably a rough approximation.

Decreasing- versus constant-height model
For mayonnaises, the decreasing- and constant-height model yield similar values for the calculated shear stress (cf. Figs. 2.1a and 2.2a). This similarity between the shear stresses is less pronounced for custards. Furthermore, for both types of products, the prediction of perceived thickness from the shear stress calculated by the constant-height model is as good as, or even slightly better than that from the decreasing-height model. The squared correlation coefficients of the semilogarithmic fit of both models are similar for custards (0.87). For mayonnaises, the squared correlation coefficient is slightly higher for the prediction by the constant-height model (0.92 versus 0.91). This suggests that the compression movement included in the decreasing-height model is not important for thickness perception and that the constant-height model is sufficient to predict perceived thickness.

To investigate the similarity in shear-stress values of both models for mayonnaises, the two terms of the decreasing-height model (Eq. 2.3), reflecting the contribution of the lateral movement and the contribution of the compression movement respectively, were compared. A calculation of these two terms (results not shown) showed that for most mayonnaises the contribution of the lateral movement was much larger than the contribution of the compression movement. However, for almost all custards the contribution of the lateral movement was smaller than that of the compression movement. This behavior is also shown in Fig. 2.3, where the calculated height of the fluid layer (Eq. 2.3) at the thickness assessment time ($t = 0.3$ s) has been plotted versus the perceived thickness for each mayonnaise and custard. This height does not depend on shear rate or velocity. The calculated height at 0.3 s deviates less from the initial height of 0.5 cm with increasing perceived thickness. For the majority of the mayonnaises, the height of the fluid layer between tongue and palate does not change significantly during oral processing, which means that according to the model, almost no compression movement occurs. For custards, a compression movement occurs to a much larger extent, causing a larger difference in calculated shear stress between the decreasing- and constant-height model.

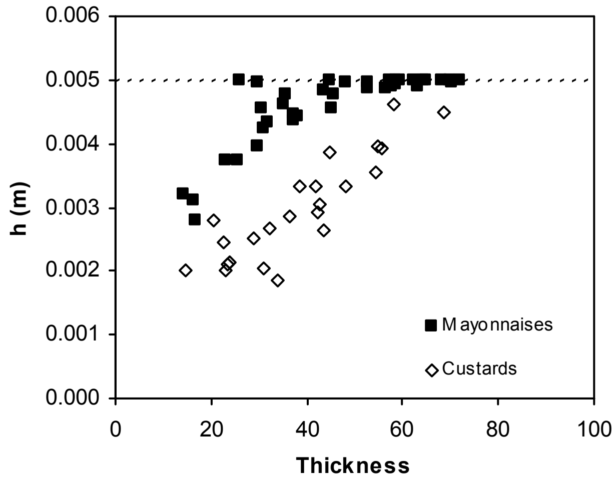

Figure 2.3 Height of the fluid layer at the end of thickness assessment time (0.3 s) versus thickness for mayonnaises and custards. The dotted line represents the initial height of the fluid layer.

Apparently, many of the mayonnaise products are too viscous to be compressed significantly in the decreasing-height model within the estimated time-span (0.3 s) and for the estimated compression force (0.25 N). We note that this observation is not consistent with real oral processing, as a compression movement is required to swallow the food product. Furthermore, the definition of thickness given by the panel also describes up-and-down motions of the tongue against the palate. Analysis of the model shows that the difference between both terms depends strongly on the choice of the physiological parameters. One of the most influential and also most disputable parameter in this study is the compression force or load exerted by the tongue. A low value (compared to literature) of 0.25 N was measured during eating of custard and assumed to be also valid for mayonnaises. It might be argued that this assumption does not hold and that the real load for the compression of mayonnaises should be higher. Dickie & Kokini (1983) estimated a value of 0.73 N for mayonnaises by squeezing a water-filled balloon attached to a syringe, and Kokini *et al.* (1977) measured values between 2.2 and 4.2 N for other types of semisolid (food) samples. Assuming a higher value for F_0 (2 N) resulted in a decrease of the calculated height at 0.3 s below the initial height for all mayonnaises (results not shown). So, with a higher value assumed for F_0, the compression movement does contribute to the shear stress of the model. The shear rate at which the decreasing-height model exhibits the best fit was lowered to 6 s^{-1} for mayonnaises. The quality of the fit, as measured by the correlation coefficient is similar to the prediction by both models at 30 s^{-1}.

However, not only the value of F_0 can cause the low extent of compression. We propose that the low extent of compression might also be attributed to the yield-stress behavior of the products, which is not incorporated in either the decreasing- or constant-height model (both models assume a power-law fluid, with two parameters, i.e., consistency K en flow index n). However, both types of products studied do show yield-stress behavior, with values ranging from 0 to 160 Pa for mayonnaises, and from 0 to 25 Pa for custards. An additional argument is that when these products are described assuming powerlaw behavior, the value for K as determined from the power-law fit is higher compared to the value obtained assuming Herschel-Bulkley yield-stress behavior (unpublished results). As K is a highly influential parameter in the model, a too high value for K implies a too low value for the compression term (cf. Eq. 2.3). This effect of a high K will affect the contribution of the compression movement more for mayonnaises than for custards, because the yield-stress values of mayonnaises are significantly larger than those of custards (see above).

Results for custards

For custards, the shear rate at which the models have the highest correlation is rather low. The value of 1 s^{-1} corresponds to a tongue velocity of 0.5 cm/s when the fluid height is estimated at 0.5 cm. Also, the correlation between thickness and shear stress for the decreasing- and constant-height model is less good than that for mayonnaises. It seems that both models in their current set-up are less well equipped to predict thickness of custards. These results seem to be independent of the choice of the physiological parameters. An important factor to explain this result for custards is the effect of saliva, which is known to break down starch. Although both types of products contain starch, the effect will be larger for custards than for mayonnaises because the consistency of custards depends mainly on the starch content, while in mayonnaises also other thickeners are present. During oral processing, saliva will mix with the food product either by gradual penetration into the food bolus and/or by oral mixing. Dilution of the product, but, more importantly, enzymatic breakdown will change the consistency and the rheological properties drastically (Engelen *et al.* 2003; Janssen *et al.* 2007). De Wijk *et al*. (2004) and Engelen *et al.* (2005) show that this breakdown of custards by amylase occurs fast enough to affect the orally perceived thickness of the food, even though thickness is perceived as one of the first attributes. The effect of saliva implies that the *in vitro* measured values for K and n are not applicable for modeling oral processing of custards. Furthermore, K and n will change in time during oral processing, and values will vary depending on the location in the food bolus.

Unrealistic values for K and n may not be the only reason that thickness of custards is less well predicted by the models. As mentioned above, also changes in oral-processing conditions or different mechanisms of perception might be involved and limit the validity of the models as the product becomes thinner. In general, custards are more fluid than mayonnaises, and this difference is significantly increased by enzymatic breakdown. This may lead to differences in physiological parameters representing oral processing or to other mechanisms for perception, i.e., the shear rate or velocity perceived in the mouth instead of shear stress, as also mentioned in the panel definition for thickness. Shama & Sherman (1973a) concluded that fluid samples (with viscosities below 0.1 Pas) are perceived at a constant shear stress instead of at a constant shear rate. In that case, thickness of fluid samples would relate to shear rate at constant shear stress, instead of the other way around. Although the viscosity of custards *in vitro* are much larger than 0.1 Pas, we expect that the considerations of Shama & Sherman (1973a) still apply because breakdown of starch by saliva could lower the viscosity significantly. By rearranging Eq. 2.2 and Eq. 2.4, tongue velocity and shear rate can be calculated at specific constant shear stresses. However, plotting thickness versus tongue velocity (obtained using the decreasing-height model) or versus shear rate (obtained using the constant-height model) does not improve the correlation of the custard data compared to the original models (results not shown).

General Considerations

In the above, we have applied existing models to our data and discussed various issues regarding our use of these models. In this section we address some general considerations regarding available models and possible new models that link rheological data with sensory data.

Oral processing

In both models (Eq. 2.2 and 2.4), tongue and palate are represented by two parallel plates. In a real mouth, however, the palate has a concave shape, the tongue can deform, and tongue and palate only touch at specific areas (Peleg 1980; Chi-Fishman *et al.* 1998; Engelen *et al.* 2002). Also, a homogeneous flow profile is assumed in the models, while in the mouth the shear deformation may be confined to a small slip layer, resulting in plug-flow. In addition, the combined lateral-compression movement of the tongue is not parallel to the palate, but is like a wedge, which moves from the front of the mouth to the back to induce swallowing (Tasko *et al.* 2002). Another comment is that the models only consider mechanical deformation and breakdown and neglect the dilution and enzymatic breakdown by saliva. As mentioned above, the effect of saliva will change the product properties drastically, especially for custards (Engelen *et al.* 2003; Engelen *et al.* 2005; de Wijk *et al.* 2004; Janssen *et al.* 2007). Furthermore, we note that viscoelastic behavior, yield stress, elongational stress and inertia effects have been neglected in the models, although they are considered relevant for oral perception (de Bruijne *et al.* 1993; Nicosia & Robbins 2001; van Vliet 2002). Viscoelastic behavior, yield stress, and elongational stress might be especially important for thick samples, e.g., mayonnaises, while inertia effects might play a role for thinner samples, e.g., custards broken down by saliva. Despite all this, both models seem adequate to describe shear stresses developed in the mouth during oral processing, especially for mayonnaises. For custards, results of both models are less good due to the omission of the effect of saliva in the models. Shear stress resulting from a combination of a lateral and a compression movement suffices to describe the mechanical deformation determining thickness perception. This is confirmed by De Wijk *et al*. (2003a), who investigated the effect of oral manipulations on the perceived thickness. It was shown that an up and down movement of the tongue, combined with shear induced by swallowing was enough to give thickness ratings comparable to normal eating behavior. More complex lateral movements, like smearing and sucking were not required for an optimal perception of thickness.

Viscoelastic behavior

Viscoelastic behavior has not been considered so far in the models used in this paper. This behavior is expected to be especially important for mayonnaises, which has a prominent yield-stress behavior due to its internal structure. Incorporation of viscoelasticity may be important for developing new and better models. Several approaches are possible to incorporate viscoelastic behavior in the modeling of orally perceived thickness.

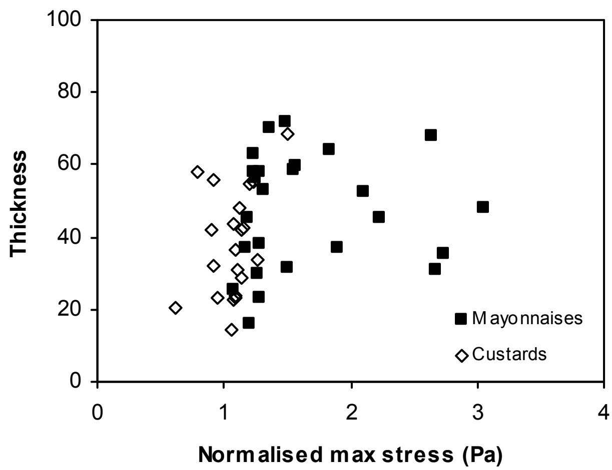

Figure 2.4 Perceived thickness versus normalized maximum stress measured during steady shear-rate measurements, for mayonnaises and custards.

Dickie & Kokini (1983) developed an improved model that accounts for the transient effects of viscoelastic materials, by assuming a relationship between perceived thickness and the normalized maximum stress of the overshoot curve (Eq. 2.5). To test this improved model for our data, thickness is plotted against normalized maximum shear stress taken directly from steady shear-rate measurements (Fig. 2.4). Figure 2.4 shows that there is no relation for either the mayonnaises or the custards. Although most of these samples do indeed show a transient peak stress, this stress does not correlate to perceived thickness, after normalization. This implies that the overshoot in stress is not the determining mechanism in the perception of thickness for our products.

During shear deformation of the sample, viscoelastic behavior also causes a normal stress, perpendicular to the tongue and palate. For one set of mayonnaises, first normal-stress differences have been recorded during flow-curve measurements. Combining shear stress and first normal-stress difference using a serial approximation (Eq. 2.6) and a harmonic approximation (Eq. 2.7) does not, however, improve the relation with perceived thickness (results not shown). This can be explained by the highly positive correlation between shear stress and first normal-stress differences in our measurements.

Normal stress felt on the tongue or palate can also contribute to the total perceived load of the tongue when pressing it up to the palate. But applying F_0 minus the normal force instead of F_0 in the decreasing-height model (Eq. 2.2) does, not change the relation between thickness and shear stress for mayonnaises (results not shown), because at low compression force (0.25 N) the contribution of the compression movement is negligible, and at a higher F_0 (2 N) the values of the normal forces are very small compared to F_0.

Conclusions

Within a limited range of shear stresses (mayonnaises < 150 Pa; custards < 30 Pa), we find a linear relationship between calculated shear stress and thickness, in accordance with the work of Kokini. Beyond this limited range, the linear relationship broke down and the thickness was found to level off with shear stress for both mayonnaises and custards. The relationship over the entire range of shear stresses can be satisfactorily described by a semilogarithmic (Fechner's) relation.

We conclude that the quality of the thickness prediction is similar for the decreasing-height model and the constant-height model, for both types of products. Furthermore, for most mayonnaises, the contribution of the lateral movement of the tongue to the shear stress in the decreasing-height model of Kokini *et al.* (1977) is orders of magnitude larger than the contribution of the squeezing or compression movement of the tongue towards the palate. This difference in magnitude is affected by the low value measured for the compression force, and by the high values for the consistency, K. The values for K were high because yield-stress behavior was neglected when the flow curves were analyzed.

For custards, the models of Kokini *et al*. (1977) were less adequate. It is proposed that this is because the models ignore interactions with saliva.
Several routes to improve the modeling by incorporating viscoelastic behavior were unsuccessful. Elongational stress and yield stress were neglected in all tested models.

In summary, we conclude that both models of Kokini *et al.* (1977) are applicable to predict perceived thickness for the semisolid food products in our study when the relation between thickness and shear stress is regarded as semilogarithmic and the effect of saliva on the product is marginal.

Acknowledgments

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3

RHEOLOGICAL BEHAVIOR, ORAL TEXTURE PERCEPTION AND THEIR RELATION FOR MAYONNAISES VARYING IN FAT CONTENT

Abstract

Relationships between texture attributes and parameters describing the rheological behavior of a set of mayonnaises (11-80% fat, limited variation in thickness) have been explored in a univariate and multivariate way. The large variation in fat content played a prominent role in the results. Sensory analysis showed that creaminess was positively related to temperature mouthfeel and melting mouthfeel and negatively to unfavorable sensations. Rheological characterization consisted of different types of small- and large deformation measurements in shear, followed by an extensive parameterization step of the curves. Parameters from dynamic stress sweeps (i.e., tan δ at 500% strain), describing structure breakdown at the start of flow correlated the best to sensory attributes, in particular to creaminess and fat-based attributes. Mayonnaises were perceived as creamier when they showed a sudden and rapid structure breakdown at the start of flow. For melting and stickiness, viscosity at high shear rates was also important. As many of the parameters and attributes were strongly affected by fat content, their relationships could be indirect instead of causal.

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Introduction

Texture can be defined as the sensory and functional manifestation of the structural, mechanical and surface properties of foods, detected through the senses of vision, hearing, touch and kinesthetics (Szczesniak 2002). For semisolid food products, orally perceived texture is expressed in a number of attributes such as creaminess, fattiness, thickness, melting and smoothness (van Vliet 2002; de Wijk *et al.* 2003b). Especially creaminess is an interesting texture attribute, as it is highly appreciated by consumers but very complex to understand (Szczesniak, 2002). Creaminess is often described as a full, velvety feeling in the mouth (Weenen *et al.* 2003).

The oral perception of attributes arises from properties of the food in the mouth, as well as on the interactions of the food and the changes of its properties during oral processing. For semisolid food products, oral processing comprises deformation and mechanical breakdown due to oral movements by the tongue and jaws, thermal equilibration, dilution and possibly enzymatic breakdown due to mixing with saliva. Food properties important for texture perception may be determined by instrumental measurements such as rheometry, which determines the rheological properties of the bulk of the food product as such or under imposed deformation. Particularly the initial properties of the product, associated with the first moment after intake of the food, are characterized in this way. It is more difficult to determine *in vitro* the changes that these properties undergo during mastication, as for this, measurement conditions should simulate conditions during oral processing, e.g., forces, flow behavior and mixing with saliva. Rheological measurements can only simulate mechanical deformation and breakdown, but to a limited extent.

Many authors (Bistany & Kokini 1983; Langton *et al.* 1999; Tabilo-Munizaga & Barbosa-Cánovas 2005) have reported on bulk rheological properties of mayonnaises. Most studies describe the rheological behavior of mayonnaises under shear, which is expressed in conventional, well-known rheological parameters such as consistency, flow index, yield stress and moduli in the linear viscoelastic region (Muñoz & Sherman 1990; Ma & Barbosa-Cánovas 1995; de Cock & Vanhemelrijck 1995; Štern *et al.* 2001; Juszczak *et al.* 2003). However, to describe the complex rheological character of mayonnaises in greater detail, more sophisticated parameters are required to quantify the different types of rheological curves (Figoni & Shoemaker 1983; Pons *et al.* 1994; Peressini *et al.* 1998).

Relating rheological properties and their changes due to imposed mechanical deformation to sensory (texture) attributes of food products can help us to increase understanding of the mechanisms behind perception. Furthermore, it can provide us with an instrumental method that can represent specific attributes and give us some insight in the oral processing conditions during perception. Studies published on the relation between texture and bulk rheological properties have focused mainly on the attribute thickness. Relations have been reported with viscosities at specific shear rates (Cutler *et al.* 1983; Daget *et al.* 1987;

Richardson *et al.* 1989), with perceived stress on the tongue (Kokini *et al.* 1977) and with overshoot stress (Dickie & Kokini 1983). For mayonnaises, relations have been found with parameters from dynamic measurements (Wendin *et al.* 1997; Tornberg *et al.* 2004). Relations between thickness and rheological properties can be very strong because both reflect bulk properties, which play a role in the bulk of the food bolus.

On the other hand, surface properties, which play a role in the outer layers of the food bolus, are not well captured by conventional rheological measurements. One of these properties is, for example friction between sample and oral tissue. Perception of attributes such as fattiness and smoothness are based on this type of property (Kokini *et al.* 1977; Malone *et al.* 2003; de Wijk *et al.* 2006b). Surface-based properties and attributes are strongly affected by the presence of fat, because fat can act as a lubricant. Although fattiness can be expected to be represented less well by bulk rheological measurements, some relations have been reported (Wendin *et al.* 1997). Fat content also plays a major role in the perception of temperature mouthfeel, which seems to reflect the heat transfer from bolus to the oral tissue (Weenen *et al.* 2003). It is an example of a bulk property that cannot be measured directly by rheometry.

Perception mechanisms underlying creaminess are multifold and hence more complicated. De Wijk *et al.* (2006a) expressed creaminess as a combination of a bulk property (describing sample consistency), a fat-related surface property (describing the interactions of the food bolus with the oral tissue) and a flavor. Another study (Kokini & Cussler 1983) showed that creaminess mouthfeel of semisolids originates from a combination of thickness and smoothness. Due to this background in viscosity- and fat-related properties, the quality of the relations of creaminess with bulk rheological parameters can be expected to be in between those for thickness and purely surface-related attributes. Relations with creaminess have been reported for viscosity and consistency (Tunaley *et al*. 1985) and for the viscoelastic moduli in the linear viscoelastic region (Clegg *et al.* 2003). Besides these linear relations, results have been reported that suggest an optimum in the relationship between creaminess and viscosity (Wood 1974; Daget *et al.* 1987). Recent studies on multivariate relations of creaminess and other texture attributes with a wide range of rheological parameters (Jellema *et al.* 2005; Janssen *et al*. 2007, 2008) have revealed correlations of creaminess with critical strain values, nonlinear viscoelastic behavior and viscosities at high shear rates.

Another relevant attribute is melting mouthfeel, or perceived melt-rate, which was often explained as the opposite of thickness. However, recent research suggests that it is a more complex attribute, reflecting the thinning of the product in the mouth and involving surface-related properties next to bulk properties (de Cock & Vanhemelrijck 1995; Janssen *et al.* 2007; de Wijk *et al.* 2006a).

The aim of this study is to describe the sensory and rheological characteristics of mayonnaises and to relate their texture attributes, especially creaminess, to an extensive set of rheological parameters in a univariate and multivariate way. Compared to previous studies, a wide range of texture attributes has been determined, as well as a wide range of bulk rheological properties under shear. The sample set consisted of commercially available mayonnaises with a wide range of fat contents and a limited variation in sensory thickness. For this reason, results will be complementary to those by Janssen *et al.* (2008) on model mayonnaises where variation in sensory thickness played a dominant role. Rheological measurements comprised small deformation (dynamic stress and frequency sweeps) and large deformation (flow curve and steady shear rate) measurements, to achieve different deformation conditions resembling those in the mouth. In a previous publication on the same data and sample set (Terpstra *et al.* 2004), texture attributes have been related to all measurement points of the rheological curves, to obtain measuring conditions that correlate maximum with specific attributes. In the current study, the rheological curves have been transformed into rheological parameters by extensive parameterization. In addition to conventional rheological parameters and parameters obtained by fitting of yield stress behavior, structure breakdown and frequency dependency, extra parameters have been introduced to describe the complex rheological behavior of the mayonnaises, e.g., viscoelastic behavior at specific strains, critical energy and concaveness of the flow curve.

Materials and Methods

Materials

Twelve commercially available mayonnaises and dressings were used in this study, see Table 3.1. The set contained five mayonnaises (67-80% fat) and seven dressings with fat contents ranging from 11 to 41%. All dressings had been marketed as mayonnaise-replacers. In the following, the terms full-fat and low-fat mayonnaise will be used to describe the mayonnaises and dressings, respectively. Due to the commercial nature of the samples, their individual composition is not exactly known, except for the information on the labels (see Table 3.1). The low-fat as well as the full-fat mayonnaises contained hydrocolloid thickening agents, except for one of the mayonnaises with 80% fat (see Table 3.1). In general, mayonnaises with less fat contained a larger amount of carbohydrate thickening agents (i.e., carbohydrate minus sugar content, see Table 3.1). Almost all mayonnaises contained egg yolk to emulsify, vinegar to acidify and sugar to sweeten the product. All products were bought at local supermarkets in The Netherlands. They were stored at 5°C prior to testing. All measurements were performed within a few days after opening the jars.

Table 3.1 Overview of the commercial mayonnaise samples and their ingredients. All information has been obtained from the product labels.

Sample	% oil on label	Table of Nutrition, per 100 g[a]				Types of thickening agents
		total oil, g (saturated oil)	total carbo-hydrates, g (sugar)	protein, g	energy, kJ	
cM1	80	80	1.4	1	3020	none
cM2	80	80 (6)	2 (2)	1	3010	guar gum
cM3	70	71 (5.5)	5 (4)	1	2740	glucose syrup, starch
cM4	70	67 (5)*	3 (3)*	1*	2550*	glucose syrup, guar gum, xanthan gum
cM5	70	68 (5)*	5 (4)*	0.5*	2610*	glucose syrup, starch, guar gum
cM6	39	41 (3)*	11 (7.5)*	0.5*	1710*	starch, glucose syrup, guar gum
cM7	35	36 (3)	11 (7.5)	0.6	1530	glucose syrup, starch
cM8	35	36 (3)	13 (7)	0.5	1560	starch, glucose syrup
cM9	29	31*	14*	0.5*	1410*	glucose syrup, starch, guar gum
cM10	25	25	14	0.5	1170	glucose syrup, starch, guar gum
cM11	20	20 (2)	12 (7)	0.6	940	starch, glucose syrup, xanthan gum, taragum
cM12	10	11 (1.5)*	19 (10)*	0.5*	730*	glucose syrup, starch, inulin, guar gum

a For some of the mayonnaises, indicated by an asterisk, the amounts are given per 100 ml instead of per 100 g.

Rheological methods

Rheological characterization was performed by four types of measurements:
flow curve (0.01-1000 s^{-1}, 15 s per point, 36 points taken logarithmically)
steady shear rate (10 s^{-1}, total duration 1 min, 200 points taken logarithmically)
dynamic stress sweep (0.1-1000 Pa, 1 Hz, 5 s per point, 41 points taken logarithmically)
dynamic frequency sweep (0.1-50 Hz, 10 Pa, 28 points taken logarithmically)

The first three measurements were carried out with a Paar Physica MCR 300 (Anton Paar Benelux, Sint-Martens-Latem, Belgium), while the latter one has been performed on a Rheometrics SR 200 (Rheometrics Scientific, Munich, Germany). Both rheometers were equipped with a 40 mm plate/plate system on which sandpaper (kor 80) was attached to prevent slip. Samples were carefully loaded on the plate with a plastic spoon to minimize structure breakdown. After lowering the upper plate to 50 µm above final height, excess of sample was removed. The upper plate was lowered to a final gap distance of 1 mm and samples were allowed to relax and acclimatize for 10 minutes at 22°C before the measurements were made. A cover was placed over the measuring system to prevent dehydration of the sample. Measurements were made in duplicate or triplicate.

Parameterization of the rheological curves was performed with help of Microsoft Excel 97 SR-2 software. Parameters calculated from the different measurements are shown in Table

3.3. Further details on the parameterization step are given in the Results and Discussion section.

Sensory methods

Sensory scores were assessed by a trained sensory panel using a quantitative descriptive (QDA) analysis protocol (Stone & Sidel 1985). The panel consisted of ten young and healthy subjects, aged between 23 and 56 years, who were selected for their above average sensory acuity. Panelists were trained in the use of sensory odor, flavor, texture and afterfeel attributes with samples similar to those later used for measurement. They were paid for their participation. Panel testing took place at the sensory facilities of TNO Quality of Life (Zeist, The Netherlands).

Samples were assessed in three 2-hour sessions preceded by a training session. All products were presented once per session in a randomized order. During the three sessions, held at the same day and time at three consecutive weeks, subjects were presented with three replicates of each sample. Subjects were seated in sensory booths with appropriate ventilation and lighting. During presentation of a sample, subjects first smelled the mayonnaise and rated odor attributes. Then, subjects took one spoonful of the sample and rated mouthfeel and flavor attributes. After swallowing, the subjects rated afterfeel and aftertaste attributes. Attributes were rated on a computer screen using a mouse and a 100-point visual analog scale anchored at the extremes. Acquisition of the panelist's responses was done by computer using FIZZ software (Biosystemes 1998, v1.20K, Couternon, France). The attributes and their definitions (see Table 3.2a, b, c) had previously been generated using a QDA-protocol and are applicable for mayonnaises as well as custards. A more detailed description of the procedures has been given by de Wijk *et al.* (2003b).

The sample set used in the sensory study included two extra products next to the samples described in Table 3.1. They were low-fat mayonnaises with 0% fat, purchased in the United States. These samples were, however, discarded in further analysis because they behaved as outliers, strongly affecting the relationships.

Data analysis

Rheological parameters were averaged over the replicates. Sensory scores for each attribute were averaged over all replicates and panel members. Relationships within the sensory attributes and within the rheological parameters were established univariately by Pearson correlations (Microsoft Excel 97 SR-2 software) and in a multivariate way using Principle Component Analysis (PCA) and Partial Least Square Analysis (PLS1 and PLS2) (The Unscrambler 7.6 SR-1, Camo Asa, Oslo, Norway). The same type of software was used to explore relations between sensory attributes, rheological parameters and ingredients. In univariate analysis, significant correlations were checked and omitted if they were caused by groups or outliers. For multivariate analysis, sensory scores and rheological parameters were mean centered, and rheological parameters were also normalized by their standard deviation.

Results and Discussion

Sensory results

Table 3.2a, b, c indicates the range of averaged scores assessed for the different attributes. As expected, the variation in thickness mouthfeel between the samples was small. Attributes that varied strongly in the studied sample set are temperature mouthfeel, melting mouthfeel, creamy mouthfeel and afterfeel, sticky mouthfeel and some unfavorable flavor and trigeminal attributes (i.e., burning afterfeel, off flavor flavor and sour flavor).

PCA results

A PCA biplot describing the relationships between all attributes is given in Fig. 3.1. The sensory attributes in the PCA can be classified into five groups based on their location and supported by their Pearson correlation coefficients and their backgrounds. The creaminess group, consisting of creamy mouthfeel and creamy afterfeel, coincides with the first principal component axis (PC1), explaining 54% of the variance. The melting group, consisting of melting and airy mouthfeel, seems to overlap with creaminess on PC2 versus PC1, but they are separate groups because melting has a large effect on PC3, in contrast to creaminess (see Fig. 3.1). In fact, in three-dimensional space, with PC2 and PC3 explaining 18 and 11% of the variance, respectively, the creaminess group is located in between the melting group and the fat-based attributes group. This is supported by the similarity in Pearson correlation coefficients of creaminess versus melting and versus fat-based attributes (both $r = 0.85$). The group of fat-based attributes consists of attributes that correlate strongly with fat content, i.e., temperature mouthfeel, fatty mouthfeel and afterfeel and some flavors. The main representant of this group, temperature mouthfeel, is a very strong indicator of fat content of the sample, as already shown previously by Weenen *et al.* (2003). Located alongside PC2 is the group of viscosity-related attributes. Thick mouthfeel and some other attributes of this group (e.g., grainy mouthfeel, powdery mouthfeel, dry mealy mouthfeel, slimy afterfeel) are found relatively close to the origin of the PCA plot due to their limited variation in the sample set (see Table 3.2a). Instead of thickness, sticky mouthfeel is the most important attribute of the viscosity-related attributes, as it varied more between the samples. Sticky mouthfeel is grouped as viscosity-related because of its strong correlation with thickness and because the bulk component of sticky mouthfeel appears to be more important than the adhesion component for mayonnaises (Dunnewind *et al.* 2004). Melting mouthfeel is not included in the viscosity-related group, because its low

Table 3.2a, b, c List of 36 descriptive terms used for mayonnaises, including definitions and their scored range. The order of the attributes per attribute category is based on the temporal order at which the attributes are perceived during mastication. Anchors range, unless mentioned, from 'very little' to 'very much'.

Mouthfeel attributes (-mo)	Definition by panel members	Scores min - max
Temperature-mo (colder – warmer)	The degree to which the product is experienced as cold. One product can feel colder in the mouth than another, despite the fact that they have the same temperature. The attribute is judged directly at the first contact with the tongue. When the product is perceived as extremely colder, it is comparable to ice-cold water and when the product is perceived as extremely warm, it seems to have a temperature of about 25°C.	29-56
Thick-mo	The thickness of the product in the mouth after taking a bite. This attribute is perceived by moving the tongue up and down against the palate. Moving up and down is important, especially for thicker products. When a product is very thin, it spreads directly throughout the whole mouth.	54-64
Airy-mo	The degree to which the product is airy. The product feels light on the tongue and is a bit foamy and it takes little effort to disintegrate. Disintegration is executed by the tongue. Whipped egg white is very airy (maximum score) and not coherent; it takes little effort to disintegrate. Airy is perceived between tongue and palate.	14-26
Grainy-mo	The degree to which the product regarding structure contains a few or a lot of grains. It is judged by rubbing the tongue against the palate.	1-6
Creamy-mo	It is a soft, full feeling in the mouth, as well as it is somewhat 'lobbig', supple and it is soft. The product is neither rough nor dry. It leaves a soft, fatty feeling. It is often a combination with fat. It is a feeling that is perceived in the whole mouth and with the whole inside of the mouth. There is a velvety feeling in the whole mouth.	36-59
Sticky-mo	Sticky feeling that can be perceived by tongue and palate. A really very sticky product, for instance, is the caramel of a candy bar. It is perceived between the teeth during mastication. The degree to which the product comes loose from different parts of the mouth determines the intensity of sticky.	27-50
Heterogeneity-mo	The feeling you get when a product has at the same time something thin and something thick. This feeling can also be described as cloudy or flocky. It arises when the product does not melt evenly, after putting it in the mouth. Sometimes, it takes a while before the feeling is perceived. This feeling is perceived in the whole mouth.	9-22
Dry-mealy-mo	Represents a dry/mealy feeling in the mouth. Difficult to swallow. It is perceived between tongue and palate. It is difficult to remove the product and it extracts saliva from the mouth. When moving the tongue through the mouth, it feels rough. You are inclined to have a drink.	13-23
Melting-mo (slow – fast)	The speed with which the product becomes liquid and spreads in the mouth. It is the gradual process of becoming thinner in the mouth. The product is also diluted with saliva and it is a term that is measured in time. The feeling is perceived between the tongue and the front of the palate.	27-53
Fatty-mo	Represents a fatty feeling in the mouth. Indicates a slippery feeling and a sealed coating on the palate. At first, it is perceived on the inside of the edges of the teeth, later also on the outside of the edges of the teeth.	40-60
Powdery-mo	The product has a structure of meal porridge and it homogeneous. The powdery feeling can be perceived between tongue and palate.	8-14
Prickling-mo	A prickling/stinging/biting feeling that is perceived on the tongue. Recall very low-carbon dioxide containing drinks or spicy food. 'Tintelen' is very lightly prickling and prickling has a higher intensity.	26-45
Astringent-mo	A chalky, rough feeling in the mouth (mainly on teeth) as if one eats nuts or spinach. It is perceived at the following locations: at the front of palate (edge of gums and teeth), in the back of the throat and at the back of the tongue and cheeks.	21-37

Afterfeel attributes (-af)	Definition by panel members	Scores min - max
Creamy-af	The degree to which the product leaves a soft/creamy feeling in the mouth. It is related to the creamy mouthfeel sensation, but the soft/creamy sensation is less intense than with creamy mouthfeel (velvety feeling).	12-37
Sticky-af	Sticky feeling on teeth and molars after expectorating the product. The degree in which the product leaves a sticky feeling in the whole mouth.	20-29
Fat-af	The degree to which the product leaves a fatty feeling. It is a typical coating of the mouth. It can be perceived on teeth and palate by sweeping it with the tongue.	22-40
Burning-af	Burning feeling that remains after having a spicy/herby/sour product in the mouth.	21-45
Astringent-af	Astringent/rough feeling in the mouth, after consuming, for instance, spinach/wine/rhubarb, which remains after expectorating. It is perceived in the whole mouth.	25-42
Slimy-af	Degree to which the product leaves a slimy feeling after swallowing. One is inclined to keep on swallowing. It is made up from thicky, stringy saliva, sticking together.	16-22
Salt-at	Salty aftertaste of the product.	16-26

Odor attributes (-od)	Scores min - max
Intensity-od	44-61
Sour-od	35-50
Vanilla-od	1-4
Herbs-od	9-17
Dairy-od	7-31
Off odor-od	17-27

Flavor attributes (-fl)	Scores min - max
Intensity-fl	56-64
Salt-fl	31-42
Sour-fl	42-62
Vanilla-fl	1-5
Oil/fat-fl	36-53
Herbs-fl	16-27
Egg-fl	11-24
Dairy-fl	6-19
Sweet-fl	11-30
Off flavor-fl	10-31

correlation with thickness and stickiness and its location in Fig. 3.1 support the view that melting is not the opposite of thickness. The last group of attributes is that of the unfavorable sensations, formed by unfavorable flavor and trigeminal attributes that have a negative effect on the perception of creaminess. Attributes of this group are negatively correlated with creaminess (max r = -0.89).

The location of the mayonnaise samples in Fig. 3.1 is indicated by their fat percentage. Their relative location in the PCA can be largely explained by their fat content. Full-fat samples are located in the upper right quadrant, scoring high on creaminess and fat-based attributes. Samples with lower fat content are spreaded over the remaining quadrants, their individual location depending on their scores on unfavorable flavor and trigeminal attributes and sweet flavor.

The PCA plot for commercial mayonnaises (Fig. 3.1) shows similarities with the ones found by Janssen *et al.* (2008) and Weenen *et al.* (2003) for model mayonnaises and for a different set of commercial mayonnaises, respectively. There are also some distinct differences, which can be attributed to the small contribution of thickness and the more prominent role of unfavorable sensations in our commercial sample set.

Prediction of creaminess
Creamy mouthfeel has been predicted from sensory attributes by multivariate PLS1 to find its underlying attributes. Creamy afterfeel was omitted from this prediction because it is a good predictor of creamy mouthfeel on its own (slope of the regression line of the measured versus predicted creamy mouthfeel = 0.90, correlation coefficient = 0.93). Creamy mouthfeel could be predicted very well (slope = 0.92, r = 0.96) by a three-fold linear combination of temperature mouthfeel, melting mouthfeel and one of the unfavorable sensations (prickling mouthfeel, off flavor flavor or burning afterfeel). A sample was perceived as very creamy when it scores high on temperature and melting mouthfeel and low on the unfavorable sensations. A good prediction with two attributes remained when temperature mouthfeel or the unfavorable sensation was left out (slope = 0.87, r = 0.92).

These PLS results support previous research that showed that creaminess of mayonnaises is more than texture alone; (unfavorable) trigeminal attributes and/or flavors are also included. Interestingly, we found melting to be a positive factor for the prediction of creaminess, instead of thickness found by other authors (Kokini & Cussler 1983; Elmore *et al.* 1999; Janssen *et al.* 2007, 2008). Fat content is also shown to be important for the perception of creaminess of mayonnaises, and can be included either by fatty mouthfeel, or by nontexture attributes, i.e., temperature mouthfeel (Weenen *et al.* 2003) or flavor (Tepper & Kuang 1996; Wendin *et al.* 1999).

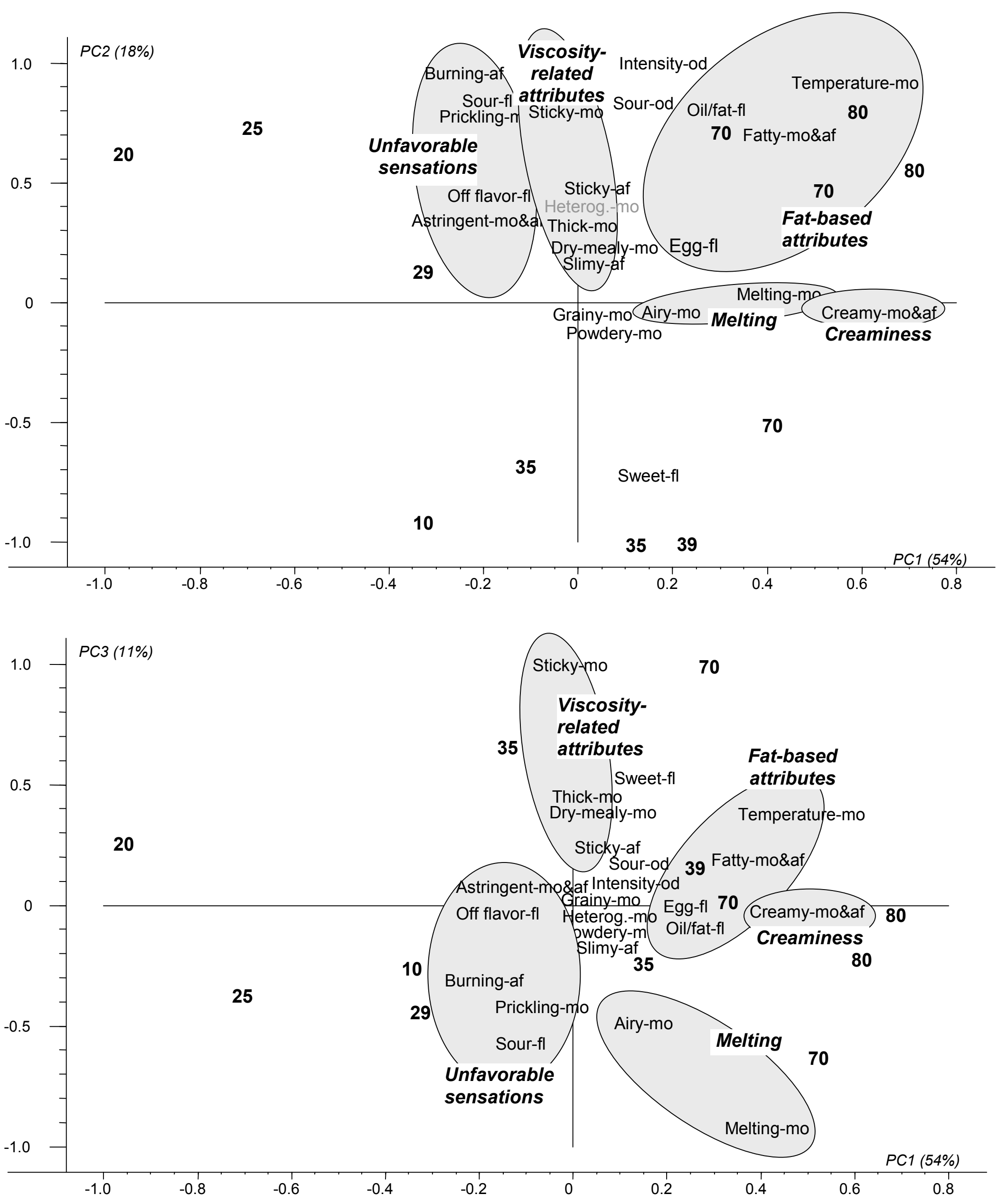

Figure 3.1 PCA biplots of sensory attributes with the relative location of the samples. The upper biplot shows PC2 versus PC1, the lower biplot PC3 versus PC1. The samples are indicated by their fat content in percentages. For the sake of clarity, some odor and flavor attributes with limited variation have been omitted and mouth and afterfeel of the same attributes that are strongly correlated have been combined. Fat-based attributes include temperature-mo, fatty-mo, fat-af, oil/fat-fl and egg-fl; viscosity-related attributes include sticky-mo, thick-mo, sticky-af and dry/mealy-mo; unfavorable sensations include burning-af, sour-fl, prickling-mo, off flavor-fl and astringent-mo and -af.

Rheological results

Typical rheological curves for the mayonnaises are shown in Fig. 3.2a, b, c, d. Table 3.3 indicates the parameters extracted and calculated from the curves, including the ranges found for the different samples. In general, the measurements were very reproducible, with standard deviations between the replicates of the obtained parameters of less than 10%.

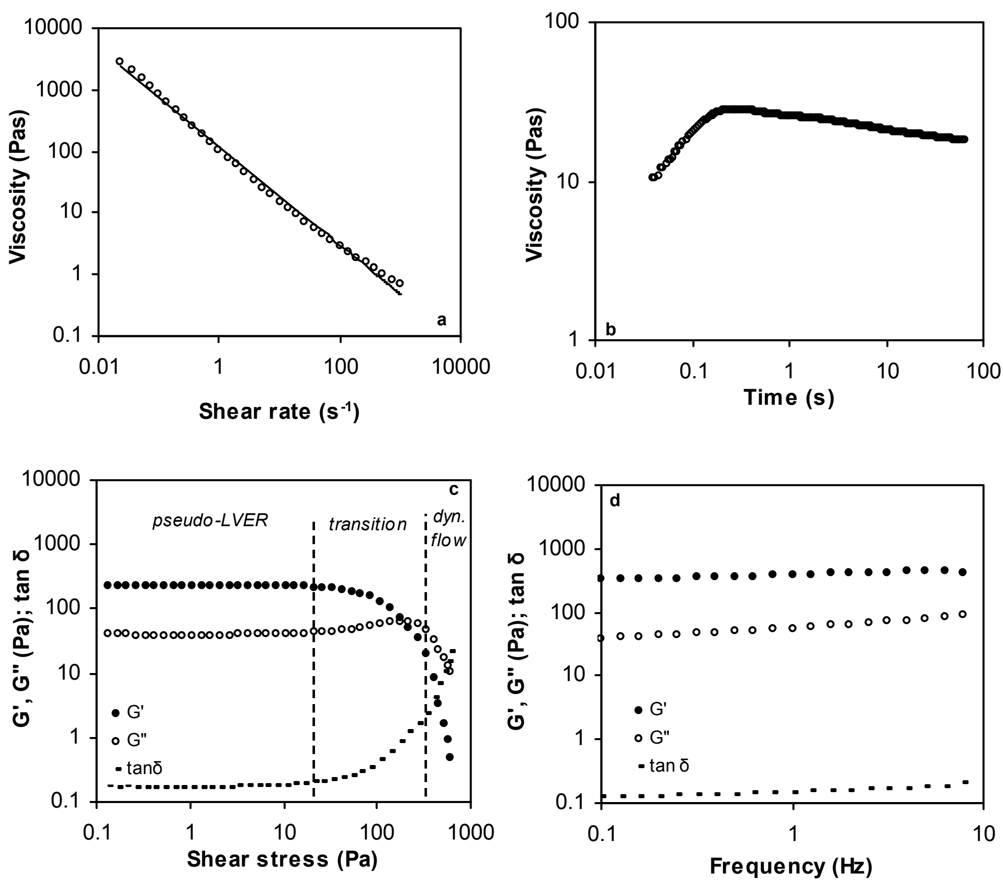

Figure 3.2a,b,c,d Typical rheological curves of the four types of measurements.

Flow-curve measurements

Flow-curve measurements of the commercial mayonnaises (Fig. 3.2a) showed a large decrease in viscosity with shear rate, indicating strong shear-thinning behavior with fairly low flow index values (see Table 3.3). Values for consistency K and flow index n correspond very well with those reported in literature for mayonnaises (Elliott & Ganz 1977; Bistany & Kokini 1983; Pons *et al.* 1994). Although for a power-law material like mayonnaises, a straight line was expected in a double logarithmic plot of viscosity versus shear rate, the curves for our set of mayonnaises were slightly concave (see Fig. 3.2a). This concaveness was expressed as C, the first coefficient of a second order polynomial fitting the double logarithmic plot (Guion & Hood 1985). C has a small but positive value for the mayonnaises studied, (see Table 3.3), which means that the decrease in viscosity was faster at low shear rates than at high shear rates. Concaveness C is correlated to the amount of carbohydrate thickeners (r = -0.81); samples with a lower amount of carbohydrates (but higher in fat content) have a more concave curve. Previous research on custards (unpublished observations) has confirmed that C is related to type and amount of starch and has shown that C can become negative as well. The presence of a yield stress was established by steady stress sweep measurements (results not shown). The values of the yield stress were obtained by fitting the flow curves with the Herschel-Bulkley model (see Table 3.3), which proved to be more adequate than the Casson model. The Herschel-Bulkley fitted values for K, n and σ_y are somewhat higher than those reported for Polish commercial mayonnaises (Juszczak *et al.* 2003), but the yield stress values correspond to those reported by others (Pons *et al.* 1994; Ma & Barbosa-Cánovas 1995; Štern *et al.* 2001).

Steady shear-rate measurement

The shear rate applied during steady shear-rate measurements was 10 s^{-1}, as this is the oral shear rate suggested in literature for semisolid food products (Shama & Sherman 1973a; Cutler *et al.* 1983). The curve (Fig. 3.2b) shows overshoot behavior; a rapid increase in (apparent) viscosity at the start of the measurement followed by a gradual decrease at longer timescales. This is the result from an initial elastic response of the samples at the start of applied deformation followed by breakdown of the structure under deformation (Figoni & Shoemaker 1981). Other results (not shown) have indicated that this structure breakdown is only to a small extent reversible. The overshoot behavior of the different samples was expressed by the time and apparent viscosity at the maximum and the difference in apparent viscosity between the maximum and the end or start of the measurement (see Table 3.3). The start of the measurement has been defined as the point where the shear rate has stabilized at the set value of 10 s^{-1} for all of the samples. Fitting the overshoot behavior with the Bird-Leider model for time-dependent viscoelastic materials (Kokini & Dickie 1981) was not successful, because the model assumes power-law behavior of the primary normal stress with shear rate, and this was not the case for our flow-curve measurements (data not shown).

Table 3.3 Overview of the parameters extracted and calculated from the different rheological measurements. The classification of the rheological parameters into groups is indicated in the first column. In the second column, the range given in brackets behind the equations indicates the range of the curve used for fitting the equation. The ranges of the calculated parameters are given in the last column.

Group	Rheological parameter	Determination		Measured range (min-max)
	Flow-curve measurement (fc-)			
Large deformation flow	K n	consistency K, from flow index n, from	$\sigma = K \cdot \dot{\gamma}^n$ (0.1-100 s^{-1})	77 - 205 Pas^n 0.09 - 0.22
	σ_y K_{HB} n_{HB}	yield stress, from consistency K_{HB}, from flow index n_{HB}, from	$\sigma = \sigma_Y + K \cdot \dot{\gamma}^n$ (0.1-100 s^{-1})	26 - 174 Pa 18 - 111 Pas^n 0.19 - 0.52
	C	concaveness, from	$\log \eta = A + B(\log \dot{\gamma}) + C(\log \dot{\gamma})^2$ (0.1-100 s^{-1}) (Guion & Hood 1985)	0.005 - 0.054
	η0.1- η500	viscosity data points taken from curve at $\dot{\gamma}$ = 0.1, 10, 25, 50, 100, 500 s^{-1}		
	Steady shear-rate measurement (ssr-)			
	G	elastic shear modulus G, from	$\sigma = G \cdot \dot{\gamma} \cdot t$ (start-t_{max}) (Figoni & Shoemaker 1981)	4 - 194 Pa
	k fdσ	decay constant k, from fitted stress difference between max and end of measurement, from	$\sigma - \sigma_{60s} = fd\sigma \cdot \exp(-kt)$ (t_{max}- 60 s) (Figoni & Shoemaker 1983)	0.058 - 0.079 19 - 140 Pa
	t_{max} η_{max}	time at overshoot maximum of curve apparent viscosity at overshoot maximum of curve		0.09 - 0.44 s 16.4 - 45.5 Pas
	η_{max-e} η_{max-b}	apparent viscosity difference between overshoot maximum of curve and t=60 s apparent viscosity difference between overshoot maximum of curve and beginning of measurement (defined as the point where the shear rate stabilizes at its set value)		3.2 - 22.7 Pas 0 - 15.3 Pas
	η0.3-η60	apparent viscosity data points taken from curve at t = 0.3, 1, 3, 10, 60 s		

	Parameter	Description	Equation	Range
	Dynamic frequency sweep (dfs-)			
Behavior at rest	G'1	fitted G' at 1 Hz, from curve		262 - 1043 Pa
	G'slope	fitted slope of G' vs. frequency f, from	$G' = G'_1 \cdot f^B$ (0.1-13 Hz)	0.06 - 0.17
	G"1	fitted G" at 1 Hz, from		49 - 181 Pa
	G"slope	fitted slope of G" vs. frequency f, from	$G'' = G''_1 \cdot f^B$ (0.1-13 Hz)	0.11 - 0.22
	tan1	fitted tan δ at 1 Hz, from		0.12 - 0.31
	tanslope	fitted slope of tan δ vs. frequency f, from	$\tan\delta = \tan_1 \cdot f^B$ (0.1-13 Hz)	-0.07 - +0.14
	G'0.1-G'10 G"0.1-G"10 tan0.1-tan10	data points of G', G", tan δ taken from curve at f = 0.1, 5, 10 Hz		
	Dynamic stress sweep (dss-)			
	G'_{LVER}	fitted G' within LVER (at 1 Pa), from		234 - 902 Pa
	$slope_{LVER}$	fitted slope of G' vs. stress within LVER, from	$G' = G'_{LVER} + slope_{LVER} \cdot \sigma$ (3-8 Pa)	-3.1 - -0.4
Transition to flow	σ_{critA}	critical stress A, stress at crossing-over point of G' and G" (exact calculation by interpolation of two nearest measurement points)		93 - 313 Pa
	γ_{critA}	critical strain A, strain at crossing-over point of G' and G" (exact calculation by interpolation of two nearest measurement points)		97 - 233%
	E_{crit}	critical energy, area under strain vs. stress curve up to the critical point		34 - 176 J/m^3
	σ_{critB}	critical stress B, stress at point where G' decreases maximally between two consecutive measurement points		79 - 293 Pa
	γ_{critB}	critical strain B, strain at this point		74 - 169%
	dG'max	maximum decrease in G' between two consecutive measurement points (see critB)		29 - 199 Pa
	dG'_{after}	difference between G' at 5 and 250 Pa		192 - 559 Pa
	dG''_{after}	difference between G" at 5 and 250 Pa		-70 - +52 Pa
Behavior at rest (0.1- ±10 Pa, 1-10%) **Transition to flow** (±10- ±350 Pa, 10-50%) **Dynamic flow** (350-1000 Pa, 500%)	G'5-G'500 G"5-G"500 tan5-tan500 strain5-strain500	data points of G', G", tan δ, strain taken from curve at σ = 5, 33, 50, 100, 250, 500 Pa (values at 500 Pa taken as close as possible to 500 Pa)		
	G'1%-G'500% G"1%-G"500% tan1%-tan500% stress1%-500%	data points of G', G", tan δ, stress taken from curve at γ = 1, 10, 50, 500% (values at 500% calculated by interpolation of two nearest measurement points)		

Alternatively, the overshoot-curve was splitted up for modeling. From the first part, until the maximum, the linear slope was calculated. As this part reflects initial elasticity of the sample, this slope equals the shear modulus G (Figoni & Shoemaker 1981). The second part of the curve, reflecting structure breakdown was modeled by an exponential stress decay model (Figoni & Shoemaker 1983), which performed better than a first order model reflecting thixotropic behavior (Tiu & Boger 1974). R-squared values of the fit with the model of Figoni & Shoemaker (1983) ranged from 0.81 to 0.98. Curves of low-fat mayonnaises could be fitted better than curves of full-fat mayonnaises. Parameters obtained from this model, i.e., the viscosity decay constant k and fitted stress difference between the maximum and the end of the measurement are given in Table 3.3.

Dynamic stress sweep measurements
In the dynamic stress sweep curves (Fig. 3.2c), three regions can be distinguished: the linear viscoelastic region (LVER) at low stresses, a transition region at mediate stresses and nonlinear viscoelastic behavior (dynamic flow) at high stresses.

At low stresses, in the LVER, the structure of the mayonnaise is unaffected by the applied stress and the viscoelastic properties of the intact sample structure are being measured ('at rest'). The storage modulus, G' and the loss modulus G" are fairly constant over this region (see Fig 3.2c). As the storage modulus G' is larger than the loss modulus G", the sample behaved mainly elastically, as a viscoelastic solid. For the mayonnaises samples in this study, the LVER is in fact a pseudo-linear viscoelastic region, because G' slightly decreases with stress. The average value and decrease of G' in the pseudo-LVER was calculated by interpolation (G'_{LVER} and $slope_{LVER}$, respectively, see Table 3.3). The decrease of G' in the LVER is correlated to the amount of carbohydrate thickeners (r = -0.89), and most of the low-fat samples have large values for $slope_{LVER}$. Values of G' and G" within the pseudo-LVER found for our commercial mayonnaises are in the same range as those measured by de Cock & Vanhemelrijck (1995) and Wendin *et al.* (1997).

At mediate stress values, a transition takes place from an unaffected structure at rest to a disrupted structure at high stress that flows. The critical stress and strain, indicating the exact location of this transition can be established in different ways, depending on the definition used. Based on practical issues and the absence of correlations with other rheological parameters and sensory data, two different methods were applied in this paper: the crossing-over point of the G' and G" curves (coded as critA) and the point where the absolute decrease in G' between two consecutive measurement points is at maximum (critB), see Table 3.3. The method of critA has also been used by Wendin *et al.* (1997) to characterize dressings and mayonnaises. Compared to her values and those reported by Muñoz & Sherman (1990), critical stress values in the current study are higher. This may be caused by our products being somewhat thicker. As an extra parameter, also the critical energy, E_{crit}, was calculated as the area under the strain versus stress curve until the critical point of critA. This parameter reflects the energy required to break up the structure from rest to flow. The maximum decrease in G' between two consecutive measurement points (dG'max), as used for critB, was also determined, as well as the decrease in G' and G" after the LVER (see Table 3.3).

At high stresses, the structure of the sample is fully disrupted and the material flows. Both moduli decreased sharply, and G" becomes larger than G', which indicates liquid viscoelastic flow behavior. Values of G', G", tan δ at 500 Pa (e.g., G'500) and at 500% strain (e.g., G'500%) were extracted to represent this part of the curve. However, it should be realized that the moduli calculated in this part of the measurement are somewhat less fundamental, because the deformation at this stage will deviate from a proper sinusoidal oscillation. Due to the sudden decrease in sample integrity, the rheometer showed difficulties in reaching the set stress values for part of the samples. For this reason, the parameters at 500 Pa were computed at the stress as close as possible to 500 Pa, with actual stress values ranging from 440-530 Pa.

Dynamic frequency sweep measurement
The effect of timescale on the viscoelastic structure of the samples was determined by dynamic frequency measurements (Fig. 3.2d). All mayonnaises under study showed a weak time-dependency, with G' and G" increasing slightly with frequency. Furthermore, G' and G" were parallel in the linear viscoelastic region of the measurement. This behavior is typical for gels, and different from e.g., random coil solutions (Richardson *et al.* 1989). Calculated slopes and fitted values at 1 Hz for G', G" and tan δ are given in Table 3.3. These values are comparable to values found in the LVER of the dynamic stress sweep and to values in literature (Wendin *et al.* 1997; Peressini *et al.* 1998).

Classification of rheological parameters
In the following of the paper, the rheological parameters obtained from the different measurement curves are classified, based on univariate correlations and their background, in four groups associated with the level of imposed deformation: at rest; transition to flow; dynamic flow; and large deformation flow (see Table 3.3). The 'at rest' group, describing the behavior of the undisturbed samples, contains all parameters from the dynamic frequency sweep measurements, as these measurements were performed within the LVER of the samples. It also contains the parameters from the dynamic stress sweep measurements within the LVER (ranging from 0.1-10 Pa and 1-10% strain approximately). The parameters 'at rest' are strongly intercorrelated, but they also show some significant correlations with rheological parameters describing the transition to flow and/or dynamic flow. For example, samples that show low G' values in the LVER, are the same as those for which the end of the LVER occurs at relatively low stress. The group of parameters describing the transition from rest to flow comprises the dynamic stress sweep parameters at mediate stresses (10-350 Pa, 10-50% strain), including the parameters describing the end

of the LVER (see Table 3.3). The group describing the behavior of the samples in dynamic flow contains dynamic stress sweep parameters taken at high stresses and strains (350-650 Pa, 500% strain). This type of flow, during small deformation oscillation, is classified separately from the flow during large deformation measurements, because correlations between parameters from both groups are less good than those within both groups.

Parameters calculated from flow-curve and steady shear-rate measurements are grouped as 'large deformation flow', because both measurements determine the flow behavior of the samples at large deformation. The parameters of these two measurements are strongly correlated to each other, but there are also some significant correlations with other groups of parameters. For example, yield stresses obtained from flow curves as well as some of the parameters describing overshoot behavior of the steady shear rate curve are strongly correlated to parameters from the 'transition to flow' group. These specific large deformation parameters describe the start of steady shear flow and are measured at strain values similar to those in the transition part of the dynamic stress sweep curve.

Relations between sensory attributes and rheological parameters

Results from univariate correlations between sensory and rheological data are given in Table 3.4. Only the most important significant correlations per attribute (group) are shown, and per attribute in the order of importance. Next to linear relations, logarithmic and power relations have been assessed as well, as previous work (Stevens 1975; Cardello 1994) suggests that psychophysical relationships are more complex than just linear relations between perceived attribute and physical stimulus. For the majority of the correlations, this approach did not result in significant improvements of the correlation coefficient. In any other case, the best correlating relation has been indicated (see Table 3.4).

Creaminess, fat-based attributes and unfavorable sensations

Univariate correlation results for creaminess and fat-based attributes (Table 3.4) overlap. Creamy mouthfeel and afterfeel are strongly correlated to rheological parameters describing behavior in dynamic flow and transition to flow. For fat-based attributes, the parameters describing behavior in large deformation flow are equally important. The strongest correlation for all these attributes was found with (the logarithm of) tan δ at 500% strain in the dynamic stress sweep measurement. Samples that flowed relatively easily at the start of flow (tanδ at 500% strain is high) were perceived as creamier and fatty and had higher scores on temperature mouthfeel. The group of unfavorable sensations can be divided in three subgroups regarding parameters and quality of correlation (see Table 3.4). The subgroup that correlates strongest with rheology shows correlations with parameters similar but opposite to those of the fat-based attributes. This indicates that the attributes in this subgroup are related to a lack of fat in the sample.

Table 3.4 Univariate Pearson correlations between sensory attributes and rheological parameters per attribute. Only the main significant ($p < 0.05$) correlations are mentioned, in the order of importance per attribute. For the sake of clarity, rheological parameters are grouped according to their rheological background (see text). Behind the rheological parameter, in brackets, the sign of the correlation is given. The string 'log' or 'pl' next to the sign indicates that the correlation is significantly better when the relationship is expressed as semilogarithmic or power law.

		max $\lvert r \rvert$	Correlating group	Specific parameter
Creaminess (mo & af)		0.84	dynamic flow	dss-tan500% (log, +)
			transition to flow	dss-dG'max (+), dss-tan50 (-)
Temperature-mo and fat-based attributes		0.93	dynamic flow	dss-tan500% (+)
			large deformation flow	ssr-tmax (-), ssr-k (+)
			transition to flow	dss-tan50 (-), dss-dG'max (+)
Melting-mo		0.86	dynamic flow	dss-G'500% (log, -)
			transition to flow	dss-tan250 (+)
			large deformation flow	fc-η500 (-)
Viscosity-related attributes	*Thick-mo*	0.76	dynamic flow	dss-G"500 (+), dss-strain500 (-)
			transition to flow	dss-G'250 (+), dss-γcritB (-)
	Sticky-mo	0.68	large deformation flow	fc-η500 (+)
Grainy-mo		0.86	large deformation flow	fc-C (-)
			dynamic flow	dss-G'500 (+)
Heterogeneity-mo		0.70	large deformation flow	ssr-fdσ (pl, +)
Unfavorable sensations	*Astringent-mo,af Off flavor-fl*	0.81	transition to flow	dss-dG'max (log, -)
			dynamic flow	dss-tan500% (log, -)
			large deformation flow	ssr-tmax (+)
	Prickling-mo Burning-af	0.75	transition to flow	dss-dG'max (log, -), dss-dG'after (log, -)
	Sour-fl	ns		
Airy-mo Powdery-mo Sticky-af Slimy-af		ns		
Other flavors		0.71	at rest	dfs-G'10 (pl, -), dss-G'LVER (-), dfs-G"slope (+)
			transition to flow	dss-dG'after (-)
Odors		0.81	large deformation flow	ssr-G (log, -)
			at rest	dss-slopeLVER (log, -)

Multivariate analysis (PLS1) of creamy mouthfeel was performed to improve the predictive power of the rheological parameters compared to univariate correlations and to see the interactions between the rheological parameters contributing to creaminess. In the several (linear) combinations of rheological parameters that were obtained, the storage modulus at 500% strain is most prominent. This is somewhat different from univariate analysis, where tan δ at the same strain performed the best. A good prediction was obtained from a combination of G' at this high strain and tan δ at low to mediate stress or frequency (50 Pa or 10 Hz). The slope of the regression line of the measured versus predicted creaminess scores is 0.71, and the correlation coefficient 0.83. This prediction could be further improved by adding a third parameter, describing the transition to flow, e.g., dG'max of the dynamic stress sweep (slope = 0.89, r = 0.93). Alternatively, a three-fold combination of the critical strain A, tan δ at mediate stress (50 Pa) and strain during dynamic flow (at 500 Pa) or tan δ at transition stress (at 250 Pa) could predict creamy mouthfeel quite good (slope = 0.87, r = 0.94). In summary, creamy mouthfeel could be predicted by a combination of parameters describing the transition to nonlinearity and describing behavior in dynamic flow.

Melting, viscosity-related attributes, heterogeneous and grainy mouthfeel

Melting mouthfeel shows strong univariate correlations with the same groups of rheological parameters as found for the fat-based attributes, but the correlations are with different parameters within the group and are less good. Samples were considered more melting when their viscosity at high shear rate (500 s^{-1}) was low and/or their storage modulus at mediate to high stress or strain was low. Thick mouthfeel and sticky mouthfeel, which have been classified in the same group of viscosity-related attributes based on sensory correlations, are different from each other with respect to their correlations with groups of rheological parameters. For thickness, G" and strain during dynamic flow are the most important parameters, as well as parameters from the transition to flow group. The relation of thickness with G" has also been found for model mayonnaises (Janssen *et al.* 2008). Sticky mouthfeel shows weak, but significant correlations with the same (groups of) parameters as thick mouthfeel, but it correlates stronger with the viscosity at 500 s^{-1} in large deformation flow. The attributes grainy and heterogeneity mouthfeel show some overlap as they are both related to parameters measured under large deformation flow. For grainy mouthfeel correlations are very strong, especially considering the low graininess scores of the samples.

Successful rheological parameters

In general, rheological parameters that correlate well with sensory attributes are those from dynamic stress sweep measurements outside the linear viscoelastic region (reflecting transition to flow and dynamic flow) and those from flow curves and steady shear-rate measurements (large deformation flow). From the three properties measured in the dynamic stress sweep, the storage modulus G' and tan δ are best related to sensory attributes, as G" only correlates with thickness. The most successful rheological parameters are the maximum decrease of G' between two consecutive measurement points (transition to flow); G' and tan δ at 500% strain and G' at 500 Pa (dynamic flow); concaveness C (from the flow curve); and the viscosity decay constant k (from the steady shear-rate measurement). So, the measurements and parameters successful in correlations with sensory attributes are those which describe the viscoelastic properties of the samples at the start of flow and its viscous behavior at large deformation flow. The critical energy parameter from dynamic stress sweep measurements has only minor correlations with sensory attributes, in contrast to results from solid food products (Szczesniak 2002; van Vliet 2002). The introduction of this parameter, reflecting the work required to go from linear to nonlinear behavior, did not improve correlations with sensory attributes compared to the already known critical strain A, because both parameters are strongly correlated.

The importance of dynamic stress sweep parameters outside the LVER in relation to sensory perception of the studied texture attributes was previously shown for mayonnaises and custards by Terpstra *et al.* (2004), Jellema *et al.* (2005) and Janssen *et al.* (2007, 2008). On the other hand, correlations of thickness with large deformation viscosity and with parameters from dynamic measurements as mentioned in literature (Dickie & Kokini 1983; Richardson *et al.* 1989; Wendin *et al.* 1997; Tornberg *et al.* 2004) were not found for the current data set. This can be due to the limited variation in sensory thickness between the samples. Štern *et al.* (2001) also found no relation between oral and rheological viscosity of model mayonnaises. Similarly, correlations of creaminess and fattiness with large deformation viscosity, flow index, consistency, yield stress and the viscoelastic moduli in the LVER, as reported by Wood (1974), Tunaley *et al*. (1985), Daget *et al.* (1987), Wendin *et al.* (1997) and Clegg *et al.* (2003) have not been found for our data.

Role of fat content

The large variation in fat content in the sample set seems to play a prominent role in the results of this study. Many attributes and rheological parameters are strongly correlated to fat content, as well as to the amount of carbohydrate thickeners, as these two are strongly confounded ($r = -0.94$). Next to the fat-based attributes ($r = 0.98$), also creaminess ($r = 0.87$), melting mouthfeel ($r = 0.71$) and some of the unfavorable sensations (max $r = -0.64$) show strong correlations to fat content. The rheological parameters that are strongly correlated to fat content include tan δ at 500% strain from the dynamic flow group ($r = 0.91$), some of the large deformation parameters describing overshoot behavior (the viscosity decay constant k, $r = 0.82$; time at max, $r = -0.71$) and some of the parameters reflecting the transition to flow (e.g., dG'max, $r = 0.76$; tan δ at mediate stress and strain, $r = -0.70$). These correlations indicate behavior in the dynamic stress sweep measurement related to fat content as shown in Fig. 3.3. Full-fat mayonnaise samples, whose structure is primarily determined by a fat-droplet network, stay firm up to high (dynamic) stresses and strains, followed by a sudden and rapid decline in structural integrity when they start to flow. Low-fat mayonnaises, where structure relies (more) on hydrocolloid thickeners, show a more gradual change in the curves at the start of flow. This difference in behavior is reflected by the values for tan δ at mediate stress and strain (a low value at 50 Pa means that the sample stays firm) and by the values for tan δ at 500% strain and the maximum decrease of G' between two consecutive points (high values mean a rapid decline). The differences between mayonnaises with high and low fat content are also displayed in the overshoot-behavior in the steady shear-rate measurements; full-fat mayonnaises have the overshoot maximum shortly after starting the measurement (t at max is low) and the following decrease in apparent viscosity is large (k is low) (see Table 3.4). It was already shown above that overshoot-behavior is related to the transition to flow behavior in the dynamic stress sweep.

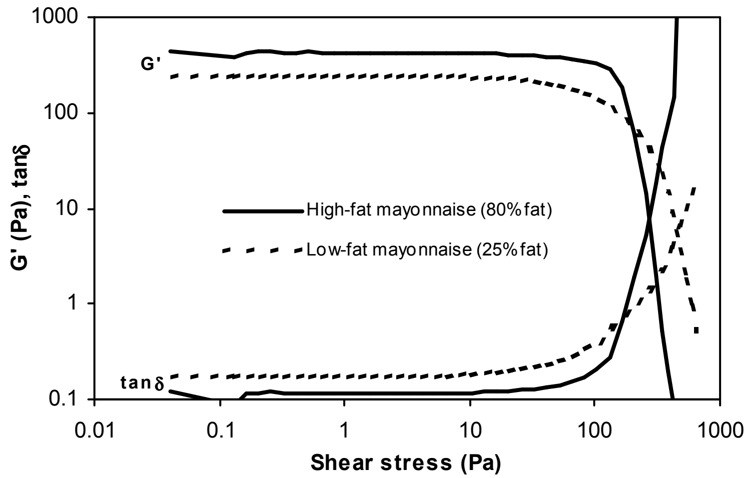

Figure 3.3 Behavior of G' and tan δ in the dynamic stress sweep measurement for a high- and low-fat mayonnaise.

The rheological parameters that are related to fat content and describe the behavior in the dynamic stress sweep and steady shear-rate measurement are the same as those that relate strongly to texture attributes. This means that the different behaviors of the mayonnaises of Fig. 3.3 can also be looked upon as the difference between mayonnaises with high and low creaminess. So, fat content and its effect on the structure breakdown at the start of flow explains the relations found between sensory attributes and the rheological parameters of the mayonnaises in this study. These relations can be causal or indirect. When the relation is causal, the structure breakdown behavior affected by fat content is directly perceived by the oral senses. Samples with high fat content are in that case rated as very creamy and fatty because it is perceived that structure breakdown at the start of flow is sudden and rapid compared to low-fat samples. The relationship between sensory perception and bulk rheology may also be indirect, via their mutual relation to fat content. In that case, fat content affects the perception of attributes via another type of underlying property, and fat content also, independently, affects the structure breakdown in bulk rheological measurements. This seems to be true for the relationship between temperature mouthfeel and rheological parameters: this attribute is perceived via the mechanism of heat transfer between bolus and oral tissue (Weenen *et al.* 2003), which cannot be measured by rheometry but is strongly affected by fat content. For a definitive assessment of the type of relation between the other sensory attributes and bulk rheological parameters, further studies should be done, which should include other potential mechanisms of perception in the measurements and/or sample set.

In the current study, surface-related attributes such as fatty mouthfeel are stronger correlated to bulk rheological parameters than thick mouthfeel and other viscosity-related attributes. Also this can be explained by the prominent role of fat content in the sample set. As shown above, bulk rheological properties can reflect surface-related attributes and attributes based on other mechanisms (e.g., temperature mouthfeel) indirectly via the effect of fat content. This is further enhanced by the small range in sensory thickness of the samples in this study compared to other studies (e.g., Janssen *et al.* 2008). Attributes that reflect surface- as well as bulk properties, such as creaminess and melting are in this study also predicted better than the viscosity-related attributes. Because of the large variation in fat content and the limited variation in sample thickness, creaminess is in the current sample set predominantly determined by surface-based properties and fat content, and thus correlates strongly with rheological parameters similar to those of the fat-based attributes. For melting mouthfeel, the actual rheological parameters that correlate best are different from those found for creaminess. Although sensory results show that melting is not the opposite of viscosity-related attributes, the rheological parameters found for melting mouthfeel show overlap with those found for the viscosity-related attributes, albeit with stronger correlations. These results indicate that melting is a very complicated attribute, relying on other mechanisms than creaminess.

Implications for oral conditions

When the relations found between sensory attributes and bulk rheological parameters are assumed to be causal, one can speculate on the oral processing conditions during texture perception. This is particularly interesting because the results in this study are not dominated by large differences in sensory sample thickness. Based on the relations found, perception of texture attributes seem to be assessed at deformation conditions in which the solid structure is starting to break down to a more fluid material. At these conditions, it is possible to distinguish full-fat structures based on fat droplet networks from low-fat structures that rely on hydrocolloid thickeners by their structure breakdown behavior at the start of flow, as described above. Melting and sticky mouthfeel correlate also strongly to viscosities at high shear rates. Similar deformation conditions were found when identifying the stresses and shear rates at which different types of rheological curves showed maximum correlation with texture attributes (Terpstra *et al.* 2004). It may indicate that these two attributes are orally perceived directly after yielding as well as during large deformation conditions at very high shear rates. Previous research has reported only lower shear rates of 1 to 100 s^{-1} for oral perception (Shama & Sherman 1973a; Cutler *et al.* 1983; Houska *et al.* 1998). When considering the correlations of creaminess, fat-based attributes and melting with parameters from the dynamic flow group, it shows that correlations with tan δ (and G') at fixed strains (i.e., 500%) are strong, while correlations with parameters taken at fixed stresses (i.e., 500 Pa) are less important. This may indicate that the oral perception of these attributes takes place at constant strain conditions. Shama & Sherman (1973a) suggest that oral perception of viscosity takes place at constant strain only for highly viscous samples (e.g., peanut butter). Comparison of the viscosity data of the mayonnaises studied here with the graph of Shama & Sherman, indicates that perception conditions of the studied products are not in the constant-strain part of the graph, but are situated in the bending point between constant shear rate and constant stress. So, the observation of perception at constant-strain conditions in our results disagrees with the graph of Shama & Sherman.

Conclusions

Sensory, rheological and mutual relations are strongly affected by the large variation in fat content and the limited variation in sensory thickness in the sample set. The sensory attributes could be classified into groups based on sensory correlations, but this is not always fully supported by their correlations with rheological parameters. Creaminess perception is related to high scores for melting mouthfeel and fat-based attributes and a lack of unfavorable sensations. Rheological measurements of the mayonnaises show complex behavior in accordance with literature; a weak viscoelastic gel, including yield stress, overshoot behavior at the start of flow and strong shear-thinning and thixotropic/rheodestructive behavior in flow. Extensive parameterization of the rheological curves was successful in introducing rheological parameters that correlate very strongly to texture attributes, i.e., tan δ at 500% strain and the maximum decrease of G' between two consecutive measurement points (both from dynamic stress sweep measurements) and the viscosity decay constant k and time of the overshoot maximum (both from steady shear-rate measurements). These parameters are strongly correlated to creaminess and fat-based attributes and describe structure breakdown at the start of flow, which is affected by fat content. Samples were perceived as creamier when they show a sudden and rapid structure breakdown at the start of flow. Such behavior is not shown by samples that were primarily thickened by non-fat thickeners. The relationship of creaminess and fat-based attributes with the mentioned rheological parameters does not have to be causal, but can be indirect via their mutual relations with fat content. Melting and stickiness are related to viscoelastic properties and viscosities at very high shear rates in large deformation conditions. From all performed measurements, dynamic measurements describing nonlinear behavior of the samples proved to be most successful in relation to sensory perception attributes.

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4

PREDICTION OF TEXTURE PERCEPTION OF MAYONNAISES FROM RHEOLOGICAL AND NOVEL INSTRUMENTAL MEASUREMENTS

Abstract

A set of commercial and model mayonnaises varying in fat content and type and amount of thickener were characterized by sensory analysis, rheological measurements and novel instrumental measurements that cover other physicochemical properties and/or reflect changes of food properties during oral processing. Predictions of texture attributes by rheological parameters were analyzed and compared to predictions by rheometry combined with novel instrumental measurements. Most of the texture attributes were predicted well by rheological parameters alone. For many attributes, such as creaminess, fattiness and melting, parameters from other instrumental measurements played a complimentary though small role. Predictions of most of the afterfeel attributes were strongly improved by the addition of parameters from the other instrumental measurements. The most important measurements in relation to texture attributes were rheological measurements at large deformation and in the nonlinear regime of the dynamic stress sweep, and two novel instrumental measurements reflecting the effect of saliva: turbidity measurements of rinse water and viscosity measurements in the structure breakdown cell with added saliva. Measurements on the initial rheological properties obtained from the LVER played no role. Relations between texture attributes and bulk rheological parameters showed that tan δ at 500% strain from the dynamic stress sweep measurement, reflecting the extent of fluid-like character of the samples during high strain dynamic flow, is the parameter that relates best to creaminess and other texture attributes. The relations between texture attributes and bulk rheological properties are similar to those of a previous study and can be regarded as generally applicable for mayonnaises.

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Introduction

The physicochemical origins of oral texture perception of semisolids have received increasing attention in the last decades (Stanley & Taylor 1993; Guinard & Mazzucchelli 1996; Wilkinson *et al*. 2000; van Vliet 2002). Despite this, the exact mechanisms underlying sensations such as creaminess, fattiness, melting and thickness are still not fully understood. Most studies have investigated relations between texture attributes (i.e., sensations) and physicochemical properties of food products. These studies consider primarily bulk rheological properties, which are reported to correlate significantly to texture attributes. It appears to be impossible though to identify a texture attribute with only one predicting rheological property or parameter. This is even true for thickness, a relatively straightforward and viscosity-related attribute (Shama & Sherman 1973). Oral perception of texture attributes seems to proceed in a more complex way; it is the result of a combination of physicochemical properties, which do not all have a bulk rheological background. Moreover, perception of texture can also be affected by perception of taste and flavors (De Wijk *et al.* 2006a). To overcome these issues, multivariate analysis and measurements other than bulk rheometry are required when studying texture perception.

Multivariate data analysis has been applied more and more next to univariate correlation in studies relating texture attributes to physicochemical properties (Tepper & Kuang 1996; Štern *et al*. 2001; Jellema *et al.* 2005; Janhøj *et al*. 2006). In the studies of Chapter 3 and Janssen *et al.* (2008), multivariate relations were explored for mayonnaises. The results showed that texture attributes could be predicted with reasonable success from rheological properties. Both studies also showed that it is important to apply different types of rheological characterization methods and that characteristics of the measured curves other than the conventional rheological properties should be considered as well. For a set of commercial mayonnaises with large differences in fat content it was shown that properties reflecting the structure breakdown of the sample at the start of flow are the most relevant for texture perception (see Chapter 3).

To further improve knowledge on the properties underlying texture attributes, measurements other than bulk rheometry are also required. These should include measurements on other physicochemical properties, as well as measurements reflecting changes in the properties of the food during oral processing. The measurements on other physicochemical properties should cover a wide range of physicochemical phenomena, including behavior of the thin film. Properties associated with this behavior, such as frictional properties, are often more fat-related than viscosity-related (de Wijk & Prinz 2005) and hence cover other aspects of perception than properties obtained from bulk rheometry.

Measurements reflecting changes of properties of the food during oral processing should determine physicochemical properties of the food bolus and the oral coating during and after mastication. When these properties are measured *in vitro*, experimental conditions should be similar to those during oral processing. For semisolid foods, oral processing is a combination of deformation and structure breakdown by oral movements, mixing with and acting of saliva and thermal equilibration. The role of saliva is multifold: it decreases the viscosity of the bolus by dilution and possibly by enzymatic breakdown of starch by α-amylase, it acts as a lubricant for the bolus because of its large salivary proteins and it neutralizes the impact of certain flavorants and tastants such as acid. Conventional rheological measurements are limited in mimicking oral processing. Small deformation rheometry determines only initial bulk properties, comparable to a food product directly after oral intake. Large deformation rheometry can mimic the mechanical deformation that is orally applied to the food bolus, albeit in a simplistic form. But for a more complete representation of the effect of oral processing, the effect of mixing with saliva should be included as well.

To complicate things further, semisolid food products do not behave as uniform and homogeneous substances in the mouth. During oral processing, the food product forms a bolus, from which the outer layers are at an earlier stage and to a larger extent affected by, for example, saliva than the inside. This means that physicochemical properties of the bulk of the bolus are different from those of the surface in contact with oral tissue (Janssen *et al*. 2007). After swallowing the food bolus, a layer of processed food product is retained, covering the oral tissue. Thin-film properties of this oral coating contribute to the perception of afterfeel attributes, which are perceived at the stage after swallowing.

Only a few researchers so far have studied physicochemical properties at mouth-like conditions in relation to perception. These studies have explored primarily *in vitro* frictional properties (Kokini *et al.* 1977; Giasson *et al.* 1997; Malone *et al.* 2003; de Wijk & Prinz 2005; de Wijk *et al.* 2006b). They have shown that these measurements are a measure of the lubricating properties of the (surface of the) food bolus and oral coating. These lubricating properties are not only strongly affected by the fat content of the sample, but also by the presence of saliva (de Wijk & Prinz 2005). Strong correlations with friction coefficients have been found for attributes associated to the surface of the bolus and oral coating, such as fattiness and fat perception (Malone *et al.* 2003; de Wijk *et al.* 2006b), as well as for smoothness, slipperiness, roughness and creamy mouthfeel (Kokini *et al.* 1977; Malone *et al.* 2003; de Wijk *et al.* 2006b).

Recently, several other novel instrumental measurements determining various types of physicochemical properties under conditions relevant to oral perception have been developed next to friction measurements (de Wijk *et al.* 2006b; Prinz *et al*. 2006; Janssen *et al*. 2007; Prinz *et al.* 2007). In many cases, specific experimental set-ups had to be designed to mimic oral conditions, making the measurements more qualitative than quantitative. One such measurement is the structure breakdown cell (Janssen *et al*. 2007), which determines the effect of saliva on the bulk viscosity of the food product. The cell consists of a helical rotating vane and fitting cup that can be attached to a rheometer. After addition of saliva to the sample in the cup, it is rapidly mixed through the bulk and subsequent changes in torque are followed in time. The effect on viscosity of mechanical breakdown, of enzymatic breakdown of starch and of dilution can be separated by comparison of measurements as such and with saliva or water added. Results on custards have shown that saliva is very efficient in breaking down the starch component, and does so in times scales relevant for oral processing. Correlations of structure breakdown cell parameters have been found with many texture attributes, including creaminess, fattiness, thickness, roughness and stickiness. Two other novel measurements can also be used to study the effect of saliva: the rate and extent of mixing of saliva into the product can be determined by *in vitro* mixing experiments in combination with image analysis (Prinz *et al.* 2007); and the overall effect of saliva on the composition of different products can be determined by measuring infra-red reflectance of *in vitro* samples (de Wijk *et al.* 2006b). Results show that this latter type of measurement relates strongly to the fat content of the products and is representative of properties of the surface of the food bolus. Composition of the oral coating retained after swallowing can be studied *in vivo* by measuring the turbidity of rinse water after swallowing the food product (Prinz *et al*. 2006). This measurement seems to reflect viscosity-related as well as fat-related properties. For custards, correlations were found with thickness, creaminess, fattiness and stickiness (Prinz *et al*. 2006).

The aim of the current study is, firstly, to test the general applicability of the relations between texture attributes and bulk rheological properties of Chapter 3 for a larger and broader set of mayonnaises and by multivariate statistical methods. Secondly, to assess whether the addition of parameters from specific novel instrumental measurements, covering other physicochemical properties relevant to oral perception, improves the predictions. The current study does not assess how well the properties obtained by the novel instrumental measurements can predict texture attributes on their own. Parameters from the novel instrumental measurements are only tested in combination with rheological parameters. Hence, the results will show whether these extra measurements are a relevant addition to bulk rheological measurements and which of them is the most useful. Furthermore, the results will give insight in oral processing conditions during oral perception of texture attributes. Rheological properties were obtained with a wide range of rheological measurements, covering different types and extents of deformation and structure breakdown: at rest, during transition to flow, in dynamic flow and in large deformation flow. The novel instrumental measurements consisted of saliva-induced viscosity changes in the structure breakdown cell (Janssen *et al*. 2007), turbidity measurements of rinse water (Prinz *et al*. 2006), *in vitro* mixing experiments of saliva in mayonnaises (Prinz *et al.* 2007) and *in vitro* infrared reflectance of mayonnaises with or without saliva (de Wijk *et al.* 2006b). Friction measurements between mouth-like surfaces (de Wijk & Prinz 2005) and fat droplet size measurements of mayonnaises as such were also included. The sample set contained commercial mayonnaises with large variations in fat content, as well as model mayonnaises with variations in, amongst others, type and amount of thickening agent. The effect of these variations in fat and in the bulk on texture attributes and physicochemical properties will be considered as well.

Materials and Methods

Materials

In total, forty commercial and model samples of mayonnaises and mayonnaise-type dressings were analyzed. In the following, the term mayonnaises will be used to describe both the real mayonnaises and the dressings. The sample set comprised four subsets, see Table 4.1. The first of these was composed of 12 commercial mayonnaises with fat contents ranging from 10 to 80%. Nearly all commercial products contained a mixture of hydrocolloid thickeners, such as (modified) starch, xanthan, guar and/or inulin. However, the exact composition of these commercial mayonnaises is not known. All commercial products were bought in local Dutch supermarkets and form a representative of mayonnaises on the Dutch market.

The other three subsets consisted of model mayonnaises, produced by TNO Quality of Life (Zeist, The Netherlands), all containing 40% fat (soy oil, Remia C.V., The Netherlands), modified starch (Farinex VA85T, AVEBE, The Netherlands) and/or xanthan (Jungbunzlauer, Austria) as thickening agent (see Table 4.1). Other ingredients were liquid egg yolk (4, 5.6 or 6.4% of the total, see Table 4.1, Nive, The Netherlands), acetic acid (3% of a 10% solution), sugar (2%), mustard powder (0.3% deactivated ground mustard powder, Tefco Food Ingredients, The Netherlands), potassiumsorbate (0.1%), ethylenediaminetetraacetic acid (EDTA) disodium (0.01%), salt (1%) and water.

Production of model mayonnaises
During production of the model mayonnaises, a 'mayonnaise' part and a 'starch' part were prepared separately. The starch part was prepared by gelatinizing the starch (10 min at 80-85°C) and then cooling it down to 20°C. The mayonnaise part was prepared by mixing the oil, egg yolk and, if any, xanthan and half of the amount of the other ingredients, followed by emulsification of the mixture in a Koruma Colloid mill provided with a rotor/stator gap of 2mm (Disho V 100/45) at 1400 rpm. The starch part and the remaining half of the other ingredients were then added to the mayonnaise part and homogenized for 5 min at 2900 rpm. Per recipe, 12 kg of product was prepared and poured into 370 ml glass jars.

Table 4.1 Fat content, amount of egg yolk, thickener composition and mean droplet size of the mayonnaise samples and the characteristics of each subset of samples. The second column indicates the sample code used in the PCA of Fig. 4.2; the commercial mayonnaises are coded by 'c' and their fat content, the model mayonnaises are coded by 'm' and the type of thickener (S = starch, X = xanthan and SX = starch and xanthan). These codes are followed by a number in case of duplicates.

	Code in PCA	Fat content	Egg yolk	Thickener phase	Droplet size (μ)	Characteristics of subset
1	c10%	10%		mixture of thickeners		Commercial mayonnaises - different mixtures of thickeners → large variation in fat content → limited variation in thickness
2	c20%	20%		mixture of thickeners		
3	c25%	25%		mixture of thickeners		
4	c29%	29%		mixture of thickeners		
5	c35%1	35%		mixture of thickeners		
6	c35%2	35%		mixture of thickeners		
7	c39%	39%		mixture of thickeners		
8	c70%1	70%		mixture of thickeners		
9	c70%2	70%		mixture of thickeners		
10	c70%3	70%		mixture of thickeners		
11	c80%1	80%		guar gum		
12	c80%2	80%		none		
13	mS1	40%	5.6%	4% starch	10.2	Model mayonnaises - starch or xanthan as thickener - variation in fat droplet size per thickener type → variation in thickness, none in fat content
14	mS2	40%	5.6%	4% starch	6.1	
15	mS3	40%	5.6%	4% starch	4.9	
16	mS4	40%	5.6%	4% starch	3.7	
17	mS5	40%	5.6%	4% starch	3.3	
18	mS6	40%	5.6%	4% starch	3.0	
19	mX1	40%	5.6%	1.3% xanthan	10.0	
20	mX2	40%	5.6%	1.3% xanthan	7.8	
21	mX3	40%	5.6%	1.3% xanthan	6.1	
22	mX4	40%	5.6%	1.3% xanthan	5.0	
23	mX5	40%	5.6%	1.3% xanthan	4.4	
24	mX6	40%	5.6%	1.3% xanthan	3.5	
25	mS7	40%	6.4%	5% starch	9.3	Model mayonnaises - starch or xanthan as thickener - variation in fat droplet size per thickener type → variation in thickness, none in fat content
26	mS8	40%	6.4%	5% starch	5.9	
27	mS9	40%	6.4%	5% starch	3.5	
28	mS10	40%	6.4%	5% starch	2.7	
29	mX7	40%	6.4%	1.3% xanthan	9.2	
30	mX8	40%	6.4%	1.3% xanthan	7.7	
31	mX9	40%	6.4%	1.3% xanthan	5.0	
32	mX10	40%	6.4%	1.3% xanthan	4.5	
33	mS11	40%	4%	5% starch		Model mayonnaises - starch and/or xanthan as thickener - variation in ratio starch versus xanthan → variation in thickness, none in fat content
34	mSX1	40%	4%	4.5% starch + 0.15% xanthan		
35	mSX2	40%	4%	4.0% starch + 0.3% xanthan		
36	mSX3	40%	4%	3.5% starch + 0.45% xanthan		
37	mSX4	40%	4%	3.0% starch + 0.6% xanthan		
38	mSX5	40%	4%	2.0% starch + 0.9% xanthan		
39	mSX6	40%	4%	1.0% starch + 1.2% xanthan		
40	mX11	40%	4%	1.5% xanthan		

The first set of model mayonnaises consisted of 12 mayonnaises prepared with modified starch or xanthan as thickening agent and varying fat droplets sizes, see Table 4.1. Differences in fat droplet size were obtained by varying the amount of egg yolk present during the homogenization step. Afterwards, the total amount of egg yolk was equaled over the samples by gently mixing in the remaining egg yolk. A second set of 8 model mayonnaises was similar in design and preparation, but with slightly different amounts of starch and total egg yolk. The third subset of 8 model mayonnaises varied in the ratio of modified starch versus xanthan, ranging from pure starch and no xanthan to vice versa in eight steps (see Table 4.1).

All mayonnaises were stored at 5°C prior to testing.

Sensory methods

Each of the four subsets of mayonnaises was tested separately by a trained sensory panel, using a quantitative descriptive (QDA) analysis protocol (Stone & Sidel 1985). The panel consisted of ten young and healthy subjects who were selected for their above average sensory acuity. Panelists were trained with samples similar to those later used for measurement and were paid for their participation. Emphasis in the training was on mouthfeel and afterfeel attributes and less on odor and flavor attributes. Panel testing took place at the sensory facilities of TNO Quality of Life (Zeist, The Netherlands).

Each subset of mayonnaises was assessed in three 2-hour sessions preceded by a training session. All products were presented once per session in a randomized order. During the three sessions, held at the same day and time at three consecutive weeks, subjects were presented with three replicates of each sample. During presentation of a sample, subjects first smelled the mayonnaise and rated odor attributes. Then, subjects took one spoonful of the sample and rated mouthfeel and flavor attributes. After swallowing, the subjects rated afterfeel and aftertaste attributes. Attributes were rated on a computer screen using a mouse and a 100-point visual analog scale anchored at the extremes. Acquisition of the panelist's responses was done by computer using FIZZ software (Biosystemes 1998, v1.20K, France). The attributes and their definitions (see Chapter 3) had been previously generated using a QDA-protocol and are applicable for mayonnaises as well as custards. More details on the sensory methods have been given by de Wijk *et al.* (2003b).

Rheological methods

Conventional rheological characterization consisted of three types of shear measurements at small and large deformation; dynamic stress sweep (0.1-1000 Pa, 1 Hz, 5 s per point), steady shear rate (10 s^{-1}, total duration 1 min) and flow curve (0.01-1000 s^{-1}, 15 s per point). All measurements were carried out with a Paar Physica MCR 300 (Anton Paar Benelux, Sint-Martens-Latem, Belgium), equipped with a 40 mm plate/plate system to which a layer of sandpaper (kor 80) was placed to prevent slip. Gap distance was 1 mm and samples were allowed to relax and acclimatize for 10 minutes at 22°C before the measurements were made. Measurements were made in duplicate or triplicate.

From the rheological curves, characteristic rheological parameters were calculated as described in Chapter 3 using Microsoft Excel 97 SR-2 software. The large set of parameters obtained in this way was reduced to 23 parameters by selection based on their univariate correlation with texture attributes and overlap between similar parameters. Parameters that did not differ between the samples in a subset (tested by Anova, $p < 0.05$) and parameters governed by the value of an outlier were discarded.

The selected parameters (see Table 4.2) represent four types of rheological properties, characterized by increasing levels of applied deformation (see also Chapter 3). Rheological properties of the undisturbed samples ('at rest') were obtained at small oscillating deformation as applied in the initial part of the dynamic stress sweep measurement. This group is represented by the storage modulus (G'), loss modulus (G") and tan δ obtained from the Linear Visco Elastic Region (see Table 4.2). A second group of parameters describes the behavior of the samples in transition from rest to flow, when the initial structure is disrupted and forced to flow by the increase in applied oscillating deformation. Parameters in this group were obtained from the dynamic stress sweep measurement at mediate stresses and include the critical stress and critical strain, indicating the end of the Linear Visco Elastic Region (see Table 4.2). Further increasing the stress in the dynamic stress sweep measurement causes the sample to 'flow' under large, oscillating deformation. The group of rheological properties measured during this regime of 'dynamic flow' are represented by G' and tan δ at 500% strain (see Table 4.2). These properties are different from flow properties measured in continuous large deformation. These latter deformation conditions were attained in steady shear-rate measurements and flow-curve measurements. Parameters representing properties in large deformation flow include the rate of (pseudo-) thixotropic structure breakdown in steady shear-rate measurements, i.e., the viscosity decay constant k (see Figoni & Shoemaker 1983). From flow-curve measurements, the viscosity at 500 s^{-1} and the concaveness of the flow curve C (Guion & Hood 1985) was obtained (see Table 4.2).

Novel instrumental methods

Friction between mouth-like surfaces

Lubricating properties of the mayonnaises, with and without saliva were measured *in vitro* using a friction tester (Halling 1976). This apparatus determined friction between a rubber band and a rotating metal cylinder onto which the sample was applied. The coefficient of friction was determined for mayonnaises as such (fr-control) and for a mixture of mayonnaises and 15% w/w saliva (fr-sal), all in triplicate. More details on the friction measurement have been given by de Wijk & Prinz (2005).

Turbidity of rinse water
Properties of the oral coating retained after swallowing were assessed by measuring the turbidity of rinse water in a custom-made photometer, as described by Prinz *et al.* (2006). Following sensory assessment, subjects swallowed a spoonful of mayonnaise whereupon they immediately rinsed with 20 ml of water for 5 s before spitting the rinse water back into a container. The turbidity of this first rinse water is coded as turb-R1. Subjects then rinsed once more in the same manner (turb-R2). The decrease in turbidity between the first and second rinse is coded as turb-change. Both parameters reflect the composition (fat content, thickener) of the sampled oral coating (de Wijk *et al.* 2006b).

Effect of saliva on infra-red reflectance
In vitro measurements of the IRR (Infra-red reflectance) of mayonnaise samples with and without saliva were carried out in a reflectometer. Measurements on mayonnaises as such are coded as irr-control, and on mayonnaises after the action of stimulated saliva as irr-sal. The difference between irr-control and irr-sal is coded as irr-change. These parameters are related to the fat phase of the product and the effect of saliva on this fat phase (de Wijk *et al.* 2006b). More details on the infra-red reflectance measurement have been given by de Wijk *et al.* (2006b).

Effect of saliva on viscosity
To investigate the effect of dilution and breakdown of the starch by saliva on sample viscosity, a recently developed structure breakdown cell (SBC) was used (Janssen *et al*. 2007) that is able to mix added liquid rapidly through the bulk. The helical rotating vane and fitting cup were attached to a standard shear rheometer (Paar Physica MCR 300). At the start of the measurement, 1 ml water or freshly collected stimulated saliva was added to the surface of the sample. Next, the vane started rotating at a constant speed of 1000 rpm for 60 s and torque values were measured. Measurements were performed at 20°C and repeated at least once. For every mayonnaise, a curve was measured as such (sbc-A, mechanical deformation only), with water (sbc-W, mechanical deformation and dilution) and with saliva (sbc-S, mechanical deformation, dilution and enzymatic breakdown of starch), see Fig. 4.1a. More details on the SBC measurement have been given by Janssen *et al*. (2007).

From the three curves, several parameters were calculated: the torque after 60 s, the decrease in torque between 0.1 and 60 s and the slope of the decrease in torque between 0.1 and 1 s and 30 and 60 s (see Fig. 4.1a). Additionally, curves were subtracted from one another to highlight specific effects; the total effect of saliva was expressed by the as such minus saliva curve (sbc-AS), the effect of enzymatic breakdown by the water minus saliva curve (sbc-WS) and the effect of dilution by the as such minus water curve (sbc-AW), see Fig. 4.1b. From these three subtracted curves the torque after 60 s and the slope of the decrease in torque between 30 and 60 s were calculated (see Fig. 4.1b).

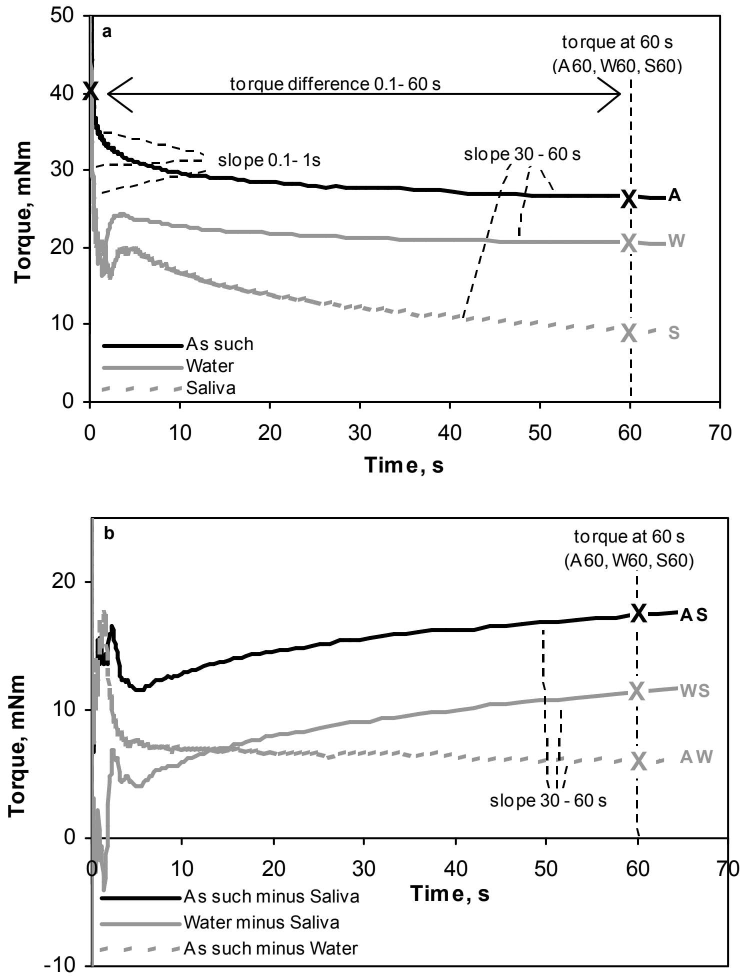

Figure 4.1a,b Examples of curves obtained by Structure Breakdown Cell measurements. (a) Measured curves as such, with water and with saliva. (b) Curves obtained after subtraction. The parameters obtained from the different curves are indicated as well.

In vitro *mixing with saliva*

The rate of mixing of saliva in the mayonnaise samples was assessed by *in vitro* mixing measurements (mixvt-) in a custom-made apparatus (Prinz *et al.* 2007). Black ink was used to color the saliva before addition to the sample. Mixing at the upper surface of the sample was recorded by a video camera. Image analysis provided two types of parameters expressing the extent of mixing: the averaged contrast between all pixels and the summed edges of phases with color differences. Instead of absolute values for contrast and edges, the standard deviation for contrast (sdc) and for edges (sde) was used to relate to sensory results, because they are more reproducible. Sdc and sde were determined for 0, 1, 2, 3, 4 and 5 s of stirring. More details on the *in vitro* mixing measurement and image analysis have been given by Prinz *et al.* (2007) and de Wijk *et al.* (2006b).

Fat droplet size
Average size of the fat droplets in the mayonnaises as such was measured with a Coulter Laser LS230 (Coulter Electronics, Mijdrecht, The Netherlands). Samples were prepared by suspending 1.5 g of mayonnaise in 20 ml of a 0.01M SDS (Sodium Dodecyl Sulphate 2,8838 g/l) solution and gently stirred until all lumps had disappeared. The mayonnaise suspension was then centrifuged (Table centrifuge CWS 4235, 3000 rpm), and the top layer used for the fat droplet size measurement. The mean of the measured fat droplet size distribution was taken as a parameter.

Data analysis and prediction

Sensory scores for each attribute were averaged across panel members and replicates. For the present study, only results for texture attributes were used (see Table 4.3 and 4.4), discarding those for odors, flavors and mouthfeel and afterfeel attributes based on trigeminal sensations. Rheological parameters and parameters from the other instrumental measurements were averaged over all replicates.

Table 4.2 Selected parameters and the rheological measurement in which they were obtained, per group. More details on the background of the selected parameters have been given in Chapter 3.

Group	Type of measurement	Selected parameters
Properties 'at rest'	dynamic stress sweep (dss-)	- G', G" and tan δ at 1 Pa
Properties during transition to flow	dynamic stress sweep (dss-)	- G', G" and tan δ at 50 Pa - G', G" and tan δ at 250 Pa - critical stress and critical strain (cross-over point of G' and G") - critical ratio (ratio of critical strain and critical stress)
Properties in dynamic flow	dynamic stress sweep (dss-)	- G' and tan δ at 500% strain
Properties in large deformation flow	steady shear rate (ssr-)	- η at maximum of overshoot curve - η at 1 and 60 s - exponential viscosity decay constant k
	flow curve (fc-)	- η at 1, 100 and 500 s^{-1} - power law flow index n - concaveness C

Multivariate relationships among and between texture attributes and parameters from rheometry and other instrumental measurements were established by Partial Least Square Analysis (PLS2) and Principal Component Analysis (PCA) (The Unscrambler 7.6 SR-1, Camo Asa, Norway). In multivariate analysis, all data were mean centered and parameters from rheometry and other instrumental measurements were normalized by their standard deviation. Validation of the models obtained by PLS2 was performed by cross-validation with a fixed segmentation of 10 segments, containing 4 samples each. Instead of random selection, a fixed distribution of the 40 samples over 10 segments, the same in all analyses, was chosen to ensure that within each cross validation step the remaining samples for modeling remained representative for the complete dataset. Samples were manually distributed over the segments such that samples from the same subset and/or the same location in the PLS2 and PCA score plots were equally divided over the segments. To ensure that single samples did not have major influence on certain segments, quality parameters such as leverage and residual variation were examined for each model built. After prediction and validation, a Jackknife procedure (Martens & Naes 1989) was applied to identify insignificant contributing parameters. For each variable, a variability of the regression coefficient was estimated. A variable was selected as insignificant when its absolute value was smaller than twice the standard deviation as estimated by the Jackknife procedure.

To test whether the four subsets of samples could be combined into one data set for further analysis, PCA analysis was performed on texture attributes and on rheological results. Residuals of the PCA of the combined data set were compared to residuals of PCA's in which each of the subsets in turn acted as test set. These results, as well as the overlap in values of texture attributes and rheological parameters between the subsets confirmed that the subsets could be joined into one large data set.

Results

Relations among texture attributes

The relations among texture attributes and the relative position of the mayonnaise samples have been summarized in a Principal Component Analysis (PCA) graph, see Fig. 4.2. The main sensory dimension coincides with the first principal component axis (explaining 59% of the variance) and runs from mouthfeel and afterfeel of creamy and fatty to heterogeneity. The second dimension is formed by melting, diagonally opposite grainy. The other diagonal dimension is formed by thick versus airy. Creaminess is strongly univariately correlated to fattiness ($r = 0.86$), heterogeneity ($r = -0.77$) and melting ($r = 0.75$). All model mayonnaises containing xanthan are located on the heterogeneity side of PC1. This is confirmed by a strong correlation ($r = 0.87$) between the score for heterogeneity and the presence of xanthan (instead of starch) as thickening agent. Model mayonnaises without xanthan are located closer to the creamy side of PC1, as well as close to thick. Commercial mayonnaises are all very low in heterogeneity and appear grouped according to their fat content. Full-fat mayonnaises are the creamiest samples and are located at the end of the creamy and melting axes. The other commercial products, with fat contents similar and lower than those of the model mayonnaises, are located on the creamy side of PC1, close to the nonheterogeneous model mayonnaises. Creaminess is associated with a high fat level and a low level of xanthan-induced heterogeneity.

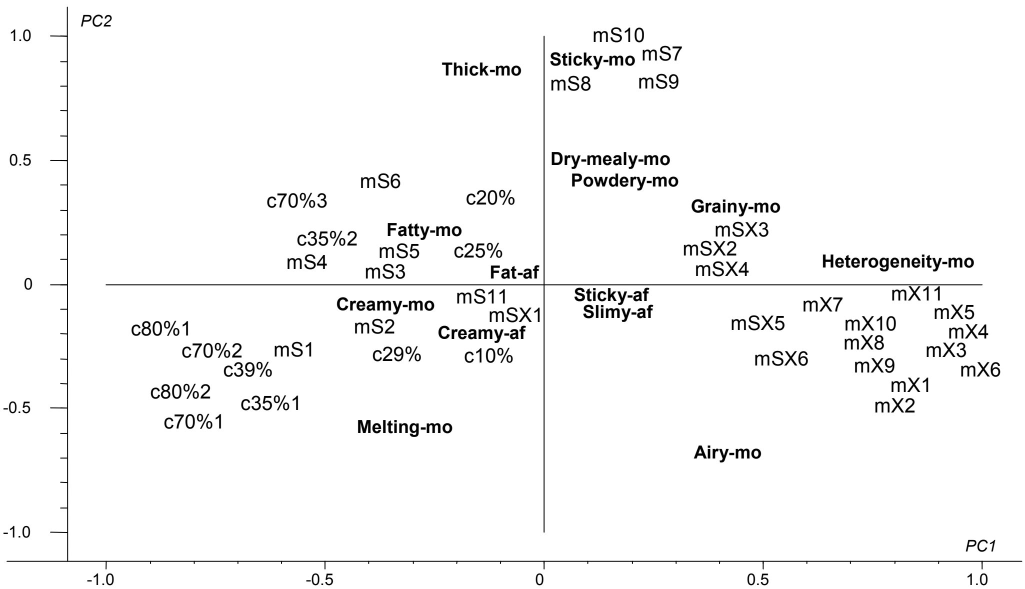

Figure 4.2 PCA biplot of texture attributes for mayonnaises. The explained variance for PC1 and PC2 is 59% and 15%, respectively. PC3 (not shown) explained 13% of the variance. The samples are indicated by their sample code, see Table 4.1 for an explanation of the code and the composition of each sample.

Predicting texture attributes from rheological parameters

Most of the texture attributes were predicted well from rheological parameters (see Table 4.3, second column). Only sticky mouthfeel and all the afterfeel attributes were predicted moderately or poorly. Heterogeneity mouthfeel was predicted very well. Groups of rheological parameters that play a role in all predictions are those that describe properties during transition to flow, properties in dynamic flow and properties in large deformation flow. The latter two groups contain nearly all parameters showing large significant regression coefficients (++ or – –, see Table 4.3). These parameters contribute strongest to the predictions of the attributes. Rheological parameters describing properties of the food product at rest are only slightly involved in a few predictions, such as those for fatty mouthfeel and fat afterfeel. The best performing rheological parameter, contributing most to many predictions, is tan δ at 500% strain of the dynamic stress sweep. This parameter, reflecting the fluid-like character of the samples during high strain dynamic flow, is

Table 4.3 Prediction of texture attributes from rheological parameters by cross-validated PLS2 models. All models were obtained by 10-fold cross-validation with fixed segments. Texture attributes that were predicted well or very well are indicated in bold. In the second and third column, the slope and correlation coefficient of the regression line of the measured attribute versus its validated prediction are indicated. From the fourth column on, the (Jackknife) significant regression coefficients of the rheological parameters in the prediction are indicated; + or -: positive or negative significant regression coefficient, ++ or --: positive or negative significant regression coefficient > 1.5.

	Quality of prediction		Properties at rest			Properties during transition to flow									Properties in dynamic flow		Properties in large deformation flow								
	slope	corrl	dss-G' 1 Pa	dss-G" 1 Pa	dss-tan 1 Pa	dss-G' 50 Pa	dss-G" 50 Pa	dss-tan 50 Pa	dss-critstress	dss-critstrain	dss-critratio	dss-G' 250 Pa	dss-G" 250 Pa	dss-tan 250 Pa	dss-G' 500%	dss-tan 500%	ssr- η_{max}	ssr- η 1 s	ssr- η 60 s	ssr-k	fc- η 1 s^{-1}	fc- η 100 s^{-1}	fc-η 500 s^{-1}	fc-n	fc-C
Creamy-mo	**0.77**	**0.84**				+		–				–	+	– –		++		–	+	– –	–		++	+	+
Creamy-af	0.52	0.61				+		–				–		– –		++		–		– –			++		
Fatty-mo	**0.75**	**0.83**	+		–	+		–					+		–	++	–	–	+	– –	–	+		+	
Fat-af	0.62	0.72	+		–	+		–					+		–	++	–	–	+	–					
Melting-mo	**0.76**	**0.84**			–				–	–	–	–			–	++	–	–			–	+	+	+	++
Airy-mo	**0.78**	**0.85**	+			+		–			+		–	++			–	–			+	–		– –	– –
Thick-mo	**0.83**	**0.88**							+		–	+	+	–			+	+	+			+		+	+
Sticky-mo	0.49	0.62						+		–		+	+			– –	+	+		++		+	– –	+	
Grainy-mo	**0.81**	**0.88**							+		+	+		+	+	–		+	+	–	+		– –	–	–
Heterogeneity-mo	**0.93**	**0.96**						–			++	+	–	++		– –	+	+			++	– –	– –	– –	– –
Dry-mealy-mo	**0.75**	**0.81**				+			+				+			+			+	– –	+				
Powdery-mo	**0.71**	**0.79**							+				+			+			++	– –	+			+	–
Sticky-af	0.16	0.27	+	+		+	+							+	–							–			–
Slimy-af	0.43	0.57							–	–				+	–								–		–

especially important for the attributes creamy mouthfeel, creamy afterfeel, fatty mouthfeel, fat afterfeel and melting. From the group of rheological parameters in large deformation flow, the (pseudo-) thixotropic viscosity decay constant in steady shear (ssr-k) and the viscosity at high shear rate (fc-η 500 s^{-1}) are the most relevant for attribute predictions. Similarly, from the group describing rheological properties during transition to flow, tan δ at 250 Pa is the main contributor to predictions. This parameter reflects the fluid-like character of the samples, similar to its equivalent at 500% strain, but at mediate values of stress and strain.

Creamy mouthfeel was predicted well by a combination of the parameters mentioned above, together with some other less important parameters from the same groups (see Table 4.3). A mayonnaise was perceived as creamy when the transition from solid to fluid behavior in the dynamic stress sweep happens very sudden and rapid (see also Fig. 4.3): at mediate dynamic stress the mayonnaise is still firm (dss-tan 250 Pa low), but at high dynamic strain it is so liquid-like that it flows easily (dss-tan 500% high). During large deformation flow though, the decrease in viscosity is limited (fc-η 500 s^{-1} high), showing a low level of (pseudo-) thixotropic breakdown (ssr-k low). The prediction of fatty mouthfeel from rheological parameters is more or less similar to that of creamy mouthfeel, although some parameters describing behavior at rest are also included. The predictions of creamy afterfeel and fat afterfeel are based on almost the same parameters as their mouthfeel counterparts, but the predictions are less good, see Table 4.3.

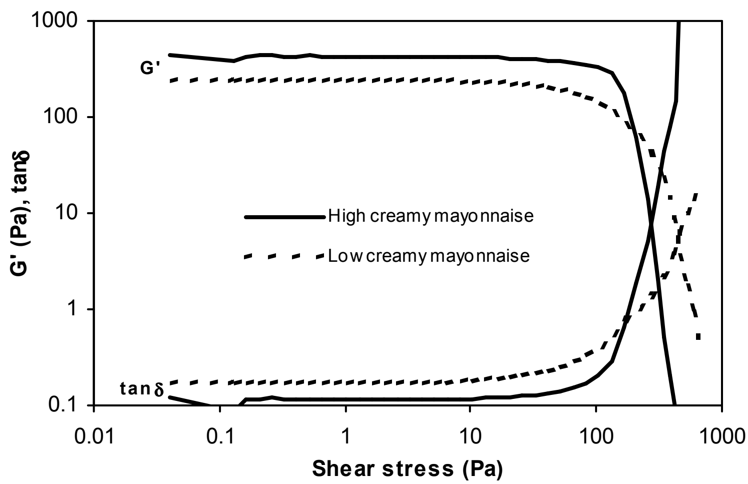

Figure 4.3 Behavior of G' and tan δ in the dynamic stress sweep measurement for a high and low creamy mayonnaise.

The combination of parameters that predicted heterogeneity mouthfeel very well represent properties during transition to flow, in dynamic flow and in large deformation flow (see Table 4.3). Many of the most relevant parameters in the prediction, i.e., dss-tan 250 Pa, dss-tan 500%, fc-η 100 s^{-1}, fc-n and fc-C are also strongly correlated to the amount of xanthan as thickening agent (not shown).

Perceived thickness was predicted well by a combination of rheological parameters describing properties during transition to flow and properties in large deformation flow (see Table 4.3). Thick mayonnaises stay firm up to mediate dynamic stresses in the transition to flow regime (tan δ at 250 Pa low, critical stress high), and subsequent viscosities in large deformation flow are high as well (viscosity at 60 s and at 100 s^{-1} high, flow index n high). For melting, parameter groups describing properties in dynamic and large deformation flow were the most relevant, whilst some parameters that describe the transition to flow played a smaller role. Mayonnaise samples that were perceived as melting behaved more fluid-like at mediate and high dynamic stresses (tan δ at 500% strain high and critical stress and strain low), but in large deformation flow the decrease in viscosity is limited (viscosity at 500 s^{-1} high and concaveness C high). Remarkably, this means that mayonnaises with weak shear-thinning behavior (flow index n close to 1) were perceived as more melting than mayonnaises with strong shear-thinning behavior. The prediction of melting is to a large extent similar to that of the strongly related attributes creaminess and fattiness. Only the behavior during the transition to flow regime is different; firmness at mediate stresses is not very important for the prediction of melting. So, in contrast to creaminess and fattiness, the transition from solid to fluid behavior does not have to be sudden and rapid for very melting mayonnaises.

Improving the prediction by adding parameters from novel instrumental measurements

Many of the texture attributes were predicted better when rheological parameters were combined with parameters from other instrumental measurements (see Table 4.4). Especially the predictions for nearly all afterfeel attributes, i.e., creamy, fat and sticky afterfeel were strongly improved, although the latter remained poor. For the other texture attributes, the improvement in quality was only to a small extent. Heterogeneity, thickness, sticky mouthfeel and slimy afterfeel did not benefit from the addition of parameters from novel instrumental measurements.

Predictions of creaminess and fattiness were improved by adding parameters from turbidity measurements of rinse water or from structure breakdown cell measurements (see Table 4.4). Both of these novel instrumental measurements consider the effect of saliva on the sample. Creamy and fat afterfeel were predicted better when turbidity parameters were added than when structure breakdown cell parameters were added. Their mouthfeel

Table 4.4 Quality of the predictions of texture attributes from all 23 rheological parameters combined with parameters from novel instrumental measurements by cross-validated PLS2 models. All models were obtained by 10-fold cross-validation with fixed segments. The slope and correlation coefficient of the regression line of the measured attribute versus its validated prediction is only indicated when adding parameters from a specific measurement clearly improved (> 0.03) the prediction based on rheological parameters alone. The last row shows which of the parameters from the novel instrumental measurement proved most important in the predictions indicated in the column. Results on the IRR, *in vitro* mixing and droplet size measurement are omitted because they did not improve the prediction of any of the attributes.

	Rheometry		+ friction		+ turbidity		+ SBC	
Number of parameters:	23		2		3		19	
Number of LV's in PLS2 model	6		6		6		5	
	slope	corrl	slope	corrl	slope	corrl	slope	corrl
Creamy-mo	0.77	0.84			0.83	0.87	0.79	0.88
Creamy-af	0.52	0.61			0.71	0.77	0.59	0.72
Fatty-mo	0.75	0.83			0.82	0.88	0.79	0.88
Fat-af	0.62	0.72			0.73	0.82	0.62	0.77
Melting-mo	0.76	0.84					0.78	0.87
Airy-mo	0.78	0.85						
Thick-mo	0.83	0.88						
Sticky-mo	0.49	0.62						
Grainy-mo	0.81	0.88	0.85	0.91				
Heterogeneity-mo	0.93	0.96						
Dry-mealy-mo	0.75	0.81			0.84	0.89		
Powdery-mo	0.71	0.79	0.74	0.82				
Sticky-af	0.16	0.27	0.21	0.34	0.23	0.36		
Slimy-af	0.43	0.57						
Important parameters			fr-c, fr-sal		turb-R1		sbc-AW60, -S60 (, -WS60)	

counterparts were similarly well predicted by adding parameters from one or the other type of measurement. When parameters of both measurements were added together to the rheological parameter set, the quality of the prediction of creaminess and fattiness did not improve further (not shown). From the three parameters obtained in the turbidity measurement, the turbidity of the water of the first rinse (turb-R1) is more important in the predictions than that of the second rinse (turb-R2), see Table 4.4. Turb-change, the difference between R1 and R2, behaves similar to turb-R1, as both are strongly correlated (r = 0.94). From the large set of parameters extracted from the structure breakdown measurements (see Fig. 4.1), only a limited number of parameters is important in the predictions of creaminess and fattiness: the measured torque of mayonnaise with added saliva after 60s of deformation (sbc-S60) and the measured torque after 60 s of the as such minus water curve (sbc-AW60). And, to a lesser extent, the measured torque after 60 s of the water minus saliva curve (sbc-WS60) and some parameters strongly correlated to these already mentioned.

The prediction of melting also benefited from the addition of parameters from structure breakdown cell measurements (see Table 4.4), with the same specific parameters as for creaminess being the most important. Grainy and powdery mouthfeel were better predicted when friction measurements were included in the predictions. Both friction parameters, with and without saliva, were equally important for the predictions, as they are strongly correlated (r = 0.90). The prediction of dry/mealy mouthfeel was strongly improved when turbidity measurements on the first rinse were included (see Table 4.4). Addition of infra-red reflectance measurements on mayonnaises with or without saliva, *in vitro* mixing measurements with saliva and droplet size measurements did not improve any of the predictions of texture attributes.

Discussion

Rheometry versus novel instrumental measurements

The predictions of texture attributes from rheometry with and without novel instrumental measurements indicate that most texture attributes of mayonnaises can be predicted well by rheological parameters alone. Addition of parameters from selected other instrumental measurements did improve the prediction for some attributes, but in most cases only to a small extent. Only for afterfeel attributes such as creamy afterfeel and fat afterfeel, addition of parameters from other instrumental measurements was required to obtain a qualitatively good prediction. The moderate predictions of afterfeel attributes by rheometry alone could be expected, as bulk behavior of the food bolus in deformation plays no role anymore after swallowing. For sticky afterfeel and slimy afterfeel, predictions were poor to moderate with and without the novel instrumental measurements added, because ratings for these attributes were only varied to a limited extent in the current study, i.e., they are located relatively close to the origin of the PCA (see Fig. 4.2).

Due to the set-up of the study, it is not possible to establish whether the parameters obtained by the novel instrumental measurements can predict texture attributes as good as or better than parameters obtained by rheological measurements. The results only indicate that there are a few instrumental measurements providing information on physicochemical properties relevant for perception that is complimentary to information obtained by conventional rheometry. The rest of the instrumental measurements provides information that is not relevant for texture perception or that overlaps with information obtained by rheometry. The two novel instrumental measurements that contribute to the perception of creaminess and fattiness indicate that information on the effect of saliva on the bulk of the bolus and on the oral coating is the most important feature lacking in conventional rheological measurements. The fact that combining turbidity measurements with structure breakdown cell measurements did not further improve the predictions seems to indicate that both measurements provide information on the same physicochemical mechanism. *In vivo* turbidity measurements reflect the composition (fat content, thickener) of the oral coating, including the effect of saliva on the composition. The first rinse, which was in this study more relevant than the second one, only takes away the outer layer of the oral coating, which is in composition comparable to the outer layer of the food bolus (Prinz *et al*. 2006). This means that the turbidity first rinse parameter reflects primarily the effect of saliva on the bulk of the mayonnaises, rather than of properties of the oral coating specifically. From the structure breakdown cell measurements, the parameters expressing the long-term effects (after 60 s) were most important for creaminess and fattiness. These parameters give an indication of the general effect of saliva (dilution as well as enzymatic breakdown) on viscosity.

In previous studies (Malone *et al*. 2003; de Wijk & Prinz 2005), friction and the effect of fat droplet size on friction were reported to play an important role for the perception of creaminess and fattiness Both factors were not found to be relevant for the mayonnaises in this study. It seems that surface properties such as friction are less important for texture perception of mayonnaises. This can be explained by the fact that friction seems to play a role only for products with low fat contents, i.e., up to 10-20% fat (de Wijk & Prinz 2005). Above this level, lubrication has reached its maximum value.

Relations between texture attributes and rheological parameters

The results show that the majority of the textural mouthfeel attributes of mayonnaises can be predicted well, though not completely, from parameters obtained by rheometry. The relations between texture attributes and bulk rheological properties can be compared to the results of Chapter 3 on commercial mayonnaises. In this latter study, the relationships found were attributed to large differences in fat content and limited differences in perceived thickness within the sample set, which dominated the sensory, rheological and interrelated results. In the current study, a larger and broader set of mayonnaises was used to assess the general applicability of the relations found in Chapter 3. Next to commercial samples, model mayonnaises with constant, mediate fat contents and different thickener phases were measured. This has resulted in a more prominent role of attributes associated with the thickener phase compared to the previous study. The attribute thickness became more important for the results, but the attribute heterogeneity became even more important. Heterogeneity was strongly correlated to the presence of xanthan as thickener and displayed a strong negative effect on the perception of creaminess. Heterogeneity was predicted very well from rheological parameters, partly due to the very large variation within the sample set. Sensory and sensory-rheological results of the current study were affected by the amount of fat in the dispersed phase as well as by the xanthan-induced heterogeneity of the thickener phase.

Despite the different characters of the sample sets involved in the current study and that of Chapter 3, the results are similar, indicating that the observed relationships between texture attributes and bulk rheological parameters can be regarded as generally applicable for mayonnaises. So, for mayonnaises, the most relevant bulk rheological measurement in relation to texture attributes is the dynamic stress sweep, especially the nonlinear regime of this measurement, from which rheological properties in dynamic flow, i.e., at 500% strain are obtained. Rheological properties describing the behavior during transition to flow and properties from both large deformation measurements also play a role in relation to texture attributes of mayonnaises. Comparison of both studies shows that properties during transition to flow and properties in large deformation flow become, respectively, less and more relevant when heterogeneity becomes important. Mayonnaise samples are perceived as creamy and fatty when the transition from solid to fluid behavior, as measured in the dynamic stress sweep measurement, is sudden and rapid and the viscosity decrease in large deformation is limited. In Chapter 3 it was shown that this behavior at the start of flow is strongly affected by the composition of the sample.

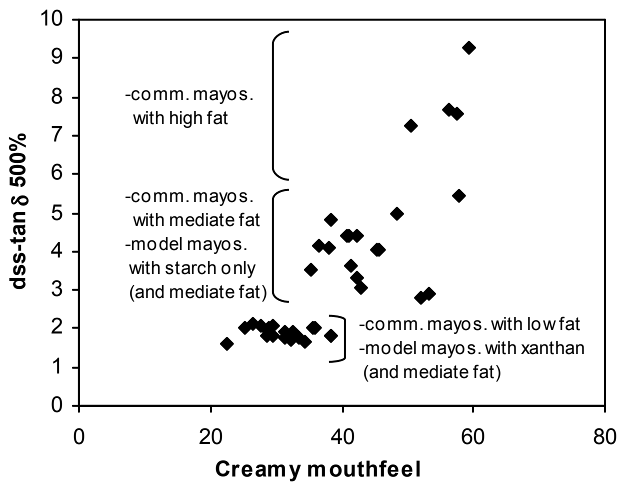

Figure 4.4 Tan δ at 500% strain from the dynamic stress sweep measurement versus perceived creamy mouthfeel. Data points for the three different groups of samples are also indicated.

The most important parameters for creaminess and fattiness are properties obtained at 500% strain of the dynamic stress sweep. In Chapter 3, tan δ at 500% strain was strongly correlated to fat content (r = 0.91). In the current study, it is correlated to fat content as well as the amount of xanthan (r = 0.74 and -0.76, respectively). This explains why tan δ at

500% strain is also in the current study successful in predicting creaminess and fattiness: this rheological parameter reflects both the effect of fat content as well as the effect of xanthan-induced heterogeneity. This is also shown when tan δ at 500% strain is plotted versus creamy mouthfeel (see Fig. 4.4). Three groups of samples can be identified in this plot, with low, mediate and high values for tan δ at 500% strain and for creaminess, respectively: commercial mayonnaises with low fat contents and model mayonnaises with mediate fat content and xanthan; commercial mayonnaises with mediate fat contents and model mayonnaises with mediate fat content and starch as sole thickener; and commercial mayonnaises with high fat contents.

Implications for oral conditions during texture perception

To translate the observed relationships between texture attributes and physicochemical properties to oral processing conditions of mayonnaises during perception of these texture attributes, it has to be established first whether these relationships are causal or indirect. When relationships are causal, a texture attribute is directly perceived by the physicochemical property that it is related to, i.e., the property represents (one of) the mechanism by which the attribute is perceived. Indirect relations between an attribute and a physicochemical property are obtained when there is a mutual relation with an ingredient or another property. In Chapter 3, it was suggested that the relationship of creaminess and other attributes with tan δ at 500% strain was indirect, because the attribute as well as the rheological parameter were strongly affected by fat content. A fat-based property, for example friction, could be the true mechanism by which creaminess is perceived. In the current study however, the same relationships between texture attributes and bulk rheological properties are established for a sample set with multiple variations in ingredients. Furthermore, several other nonrheological, fat-related properties were tested by the novel instrumental measurements, but none did improve the predictions to a large extent. For these reasons, we assume that the relations between texture attributes and bulk rheological parameters are causal and can be translated to mechanisms and conditions important for the oral perception of texture attributes of mayonnaises.

This means that bulk rheological behavior of the mayonnaise bolus is directly sensed by the oral tissue and serves as the main mechanism underlying most of the mouthfeel attributes. During oral processing of mayonnaises, deformation of the food bolus seems to be more important for the perception of textural mouthfeel attributes than the action of saliva. Furthermore, nonrheological properties, reflecting other physicochemical phenomena play a limited role in the perception of these attributes. The results suggest that oral perception of mouthfeel attributes of mayonnaises requires a certain minimal amount of deformation of the food bolus in the mouth, as initial rheological properties, measured without deformation ('at rest') were found to be irrelevant for perception. This is even the case for attributes that are perceived immediately after food intake, such as thick, airy and grainy mouthfeel. The requirement of oral movements for perception was also shown in a study of de Wijk *et al*. (2003a), in which ratings for textural mouthfeel attributes were much lower when panelists were not allowed to manipulate the food bolus with their tongue. The results of the current study suggest that the level of oral deformation required for the perception of mouthfeel attributes varies from deformation conditions at which the structure is disrupted and the mayonnaises start to flow to conditions of very large rate deformation. For the mouthfeel attributes of creaminess and fattiness, rheological behavior during these deformation conditions, as well as the effect of saliva on the food bolus is sensed by the oral sensors and translated into a creamy and fatty rating. The afterfeel versions of these attributes are perceived differently, as oral deformation is minimal during perception of these attributes. Nonrheological properties of the oral coating, as measured by the novel instrumental measurements are the main mechanism by which these attributes are perceived.

The predictions of the attributes thickness and melting indicate that these two attributes are not perceived by the same underlying mechanisms. So, they are not the opposite of each other, which was reported previously for custards (de Wijk *et al.* 2003b). This difference might be explained by the fact that saliva-induced breakdown is much more important for the structure of starch-based custards than it is for mayonnaises. For the mayonnaises in the current study, perception of melting seems more related to mechanisms similar to creaminess; oral behavior of the product at the start of flow and afterwards plays a prominent role, with a small role for the effect of saliva on the bulk properties of the food bolus. Thickness of mayonnaises seems not perceived via properties affected by saliva or physicochemical properties involved in the other instrumental measurements, but seems based solely on bulk rheological properties of the product perceived during oral movements. This is in accordance with the fact that thickness is perceived as one of the first attributes, before saliva has started to take effect.

The relationships of graininess, powderyness, dry-mealy and heterogeneity with properties measured by bulk rheometry and other instrumental measurements could be indirect instead of causal. These mouthfeel attributes are related to distinct particles, lumps, or other irregularities. The presence of particles will increase friction of the sample (de Wijk & Prinz 2005). This explains why for graininess and powderyness friction properties play a complimentary role in perception next to rheological properties in mediate and large deformation conditions. Previous studies (Malone *et al*. 2003; de Wijk & Prinz 2005) have already shown a relationship between friction and roughness, which is a related attribute. The presence of particles could be sensed directly and/or by the friction caused on the oral tissue. In case of heterogeneity, irregularities were induced by the presence of xanthan. The effect of these irregularities seems to be captured very well by some of the rheological parameters, but not by friction or any of the other properties obtained by novel instrumental measurements. This explains why heterogeneity can be predicted so well by rheometry alone, although the relations with rheological properties might not be causal.

Conclusion

Most of the texture attributes used in this study were predictable using rheological parameters only. For many attributes, such as creaminess, fattiness, melting, grainy, powdery and dry-mealy, parameters from other instrumental measurements played a complimentary role, although the improvement in the prediction was small. Predictions of most of the afterfeel attributes were strongly improved by the addition of parameters from novel instrumental measurements, because deformation, required for rheological properties is minimal during perception of these attributes. The predictions for sticky and slimy afterfeel were still poor, because these attributes varied little in the sample set. The most important measurements in relation to texture attributes are rheological measurements at large deformation and dynamic stress sweeps outside the Linear Visco Elastic Region, turbidity measurements of the rinse water of the first rinse and viscosity measurements in the structure breakdown cell with added saliva. The latter two provide information on the effect of saliva on the food product complimentary to the information obtained by conventional rheometry. Friction measurements are only useful for attributes directly related to particles. From the different parameters calculated from the structure breakdown cell measurements, the easiest to determine, i.e., after 60 s of operation, is the most important in relation to texture perception.

Relations between texture attributes and bulk rheological parameters showed that mayonnaise samples were perceived as creamier when fat content was high and/or when starch, replacing xanthan, assured a less heterogeneous product. Rheological properties reflecting the behavior at the start of flow, in dynamic flow and in large deformation flow were most relevant to the perception of texture attributes. Tan δ at 500% strain from the dynamic stress sweep measurement, reflecting the fluid-like character of the samples during high strain dynamic flow, is the parameter that relates best to creaminess and other texture attributes. These results were similar to those of Chapter 3 on commercial mayonnaises with large variations in fat content, although the sample set in the current study was broadened by the addition of samples with differences in thickener phases. This resulted in a sample set which was characterized by differences in xanthan-induced heterogeneity of the thickener phase as well as by differences in fat content of the dispersed phase. As the relations between texture attributes and bulk rheological properties were still valid for the larger and broader set of samples, they can be regarded as generally applicable for mayonnaises.

Acknowledgements

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5

EXPLORING IMPERFECT SQUEEZING FLOW MEASUREMENTS IN A TEFLON GEOMETRY FOR SEMISOLID FOODS

Abstract

The method of imperfect lubricated squeezing flow in a Teflon geometry has been explored for the characterization of elongational behavior of custards and mayonnaises. Two Newtonian products, one of low (0.07 Pas) and one of high (18 Pas) shear viscosity were used as references. Measurements of custards and mayonnaises did not behave according to either the theory of lubricated or nonlubricated squeezing flow, as there were effects of the initial sample height and compression speed. Also, calculated values for the flow index were not as we had expected. The same was true for the Newtonian samples. An important factor explaining the effect of compression speed was the presence of a certain amount of friction, rendering both lubricated theory as well as nonlubricated theory nonapplicable. Correcting for (pseudo-) thixotropic behavior of custards and mayonnaises appears to be an effective way of obtaining realistic values for the flow index. The presence of buoyancy also affected the results, especially in case of low viscous products and the effect of initial sample height. Other factors that played a role in the results were yield stress for custards and mayonnaises and instrumental artifacts associated with the imperfect set-up of the measurement, especially for the highly viscous products. Quantitatively correcting the results for all of these factors is not possible at this point. Although imperfect squeezing flow measurements in a Teflon geometry is a very practical way to measure semisolids such as custards and mayonnaises under (partly) elongational deformation, results should be regarded more qualitative than quantitative.

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Introduction

Oral texture perception of food arises from properties of the food in the mouth and the changes that occur during oral processing. For a semisolid food product, oral processing is determined by forces and velocities applied to the food product by the tongue and jaws, mixing with saliva and thermal equilibration. The combination of these factors results in deformations and structure breakdown of the food and a change in its physical properties. To get a better insight in the mechanisms of oral texture perception, we wish to measure the changing physical properties of the food product *in vitro* and relate them to its texture attributes.

Nondestructive, small deformation measurements will only yield initial product properties. To study the product properties at a later stage of oral processing, force and velocity conditions comparable to those in the mouth should be applied during the measurement, and ideally, saliva should be added. Most of the literature on rheological measurements of food only considers shear deformation. However, it seems that elongational deformation also plays a role in oral processing, next to shear deformation (de Bruijne *et al.*1993). To establish the importance of elongational deformation in oral texture perception, it is essential to measure rheological behavior of the food product in elongation.

Several methods have been developed to measure elongational behavior in semisolid and liquid products. Only a limited number of them are suitable for measuring semisolid food products with consistencies such as custards and mayonnaises. The opposing jets technique (Steffe 1996) is primarily suited to thin, liquid products, while filament stretching (Rodd *et al.* 2005) and the tubeless siphon technique (Steffe 1996) require thick, adhesive products that can undergo uniaxial tension without instant break-up. Semisolid products with intermediate consistencies can be subjected to a four-roll mill system (Macosko 1994). This technique, however is merely qualitative, as it is difficult to control and determine forces and velocities. Another set of techniques that can be used for semisolids are based on the contraction flow of material through an orifice; the pressure drop technique (Macosko 1994), Posthumus funnel (Osinga 1975; Hellinga *et al.* 1984) and forward- and backward extrusion (Steffe 1996). These techniques do not measure pure elongational behavior, because a combination of shear and elongational deformation is applied to the product. As it is not always possible to quantify the shear component within the results, most of these techniques should be considered to be empirical measurements.

In the squeezing flow technique (Chatraei *et al.* 1981; Steffe 1996; Campanella & Peleg 2002), biaxial elongational deformation is achieved by compressing the product between two parallel plates. This measurement technique has been widely used with metals, polymers and soils (Engmann *et al.* 2005), and, in the last decades, with food and other consumer products (Casiraghi *et al.* 1985; Campanella & Peleg 2002; Meeten 2002). It is probably the only nonempirical technique usable to study biaxial elongation of semisolid food products such as custards and mayonnaises. An extra advantage of the technique is that the compression movement applied resembles the movement of tongue and palate during oral processing. During the compression, shear can be present next to elongational deformation, depending on the slip conditions at the plate-product interface. Pure elongational flow can be obtained by the lubricated squeezing flow technique (Chatraei *et al.* 1981), in which slip at the plate-product interface is maximized by applying lubricants or adjusting the surface of the plates. An extensive review of fluid mechanics models in the cases of slip, no slip and partial slip conditions has been given by Engmann *et al.* (2005), for viscous, viscoplastic and viscoelastic materials. Variations within the technique include the type of compression (at constant compression force, compression speed or strain rate) and whether the product covers the area of the plates completely or not (constant area or constant volume, respectively).

A special case of the squeezing flow technique is imperfect lubricated squeezing flow in a Teflon geometry (Hoffner *et al.* 1997; Suwonsichon & Peleg 1999). Imperfect refers to the wide, shallow container replacing the bottom plate. This assures easy loading of the sample and maintains the sample without leakage during the measurement. Another advantage is the low level of damage to the sample during preparation for the measurement (Campanella & Peleg 2002). Lubrication is promoted by the Teflon surface of the container and upper plate (Suwonsichon & Peleg 1999). An important disadvantage of the technique is that the flow pattern is less well defined than in the ideal set-up, making the validity of the theoretically derived models questionable. In the last decade, imperfect lubricated squeezing flow has been used to measure the properties of a range of semisolid foods, such as mayonnaise, tomato products and yogurt (Hoffner *et al.* 1997; Lorenzo *et al.* 1997; Suwonsichon & Peleg 1999; Janhøj *et al.* 2006). From the measured data, elongational properties and the elongational flow curve of the semisolid product can be calculated. Often, a number of empirical squeezing flow parameters are extracted as well (Suwonsichon & Peleg 1999).

In the study presented here, the technique of imperfect lubricated squeezing flow in a Teflon geometry is explored for the measurement of elongational behavior of custards and mayonnaises. The aim of the study was to assess whether this technique is a practical way to determine the elongational properties of custards and mayonnaises, despite limitations due to the imperfect set-up, the use of Teflon, and the operation at high compression speeds comparable to those in the mouth. For this reason, the effect of measurement conditions (initial sample height and compression speed) were tested, in relation to the role of factors such as buoyancy, yield stress, friction and thixotropy. Next to custards and mayonnaises, a low- and high viscosity Newtonian product, that is, oil and treacle syrup, are measured to gain more insight in the method and the role of the mentioned factors. The extent of slip was studied in greater detail by applying oil as lubricant. Results are compared to those from shear measurements.

Background

Three steps can be identified in the squeezing flow measurement, see Fig. 5.1. Firstly, the sample is compressed from the initial sample height to a final sample height (in this study 1 mm). Secondly, the upper part of the geometry is halted at the final sample height to allow stress relaxation of the sample, and finally, a decompression step is applied. Figure 5.1 also shows the (empirical) parameters that are extracted from the measured curve (Suwonsichon & Peleg 1999; Corradini *et al.* 2000a): the force during compression at 2 mm sample height and at 1mm sample height (the latter is similar to the maximum compression force); and the force during the relaxation step at 1 min and at 2 min (the latter is also referred to as the residual force, R).

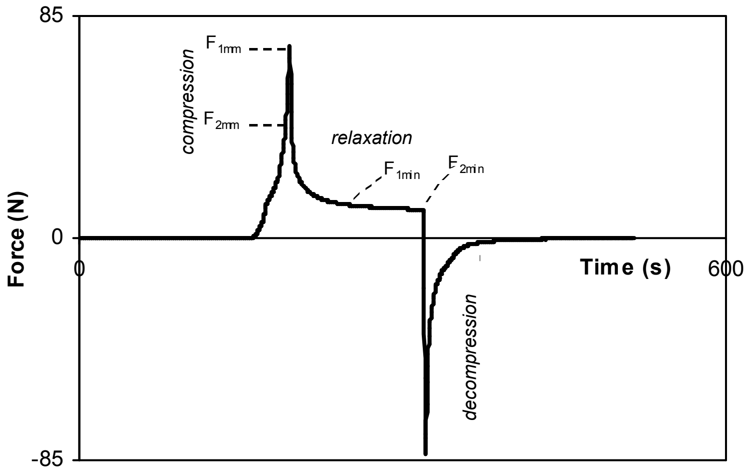

Figure 5.1 Example of a curve obtained by a squeezing flow measurement, including extractable parameters.

Data from the compression step can also be plotted as the logarithm of the force against the logarithm of the sample height (logF versus logh, see Fig. 5.2). The linear part of this curve, at low sample height, is considered to represent the region of squeezing flow, characterized by a slope and intercept. The initial part of the compression curve, on the right-hand side, is governed by instrumental artifacts and transient effects (Suwonsichon & Peleg 1999).

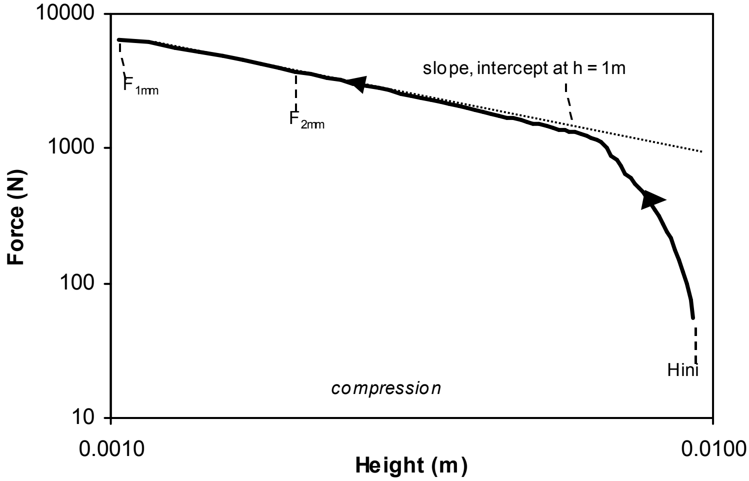

Figure 5.2 Example of a curve of logF versus logh obtained from the compression step, representative for custards and mayonnaises, including extractable parameters.

For the relation between the force and sample height during squeezing flow, several equations have been described (Campanella & Peleg 2002; Engmann *et al.* 2005). In the case of a pseudoplastic material, with consistency K and flow index n, the force during fully lubricated squeezing flow is given by:

$$F_{\text{lub}r} = 3^{\frac{n+1}{2}} \pi \cdot K \cdot R_{plate}^2 \cdot \left(\frac{V}{h(t)} \right)^n \tag{5.1}$$

and during ideal frictional flow, that is, nonlubricated squeezing flow:

$$F_{non\,\text{lub}r} = \frac{2\pi K R_{plate}^{n+3}}{n+3} \cdot \left(\frac{2n+1}{n} \right)^n \cdot \frac{V^n}{h(t)^{2n+1}} \tag{5.2}$$

where F_{lubr} is the force during lubricated compression, $F_{nonlubr}$ is the force during nonlubricated compression, R_{plate} is the radius of the upper part of the geometry, V is the compression speed and h(t) is the sample height. In case of Newtonian materials these equations simplify to:

$$F_{\text{lub}r} = \tfrac{1}{2} \pi \cdot R_{plate}^2 \cdot \mu_b \cdot \frac{V}{h(t)} \tag{5.3}$$

and

$$F_{non\,lubr} = \tfrac{1}{4}\pi \cdot R_{plate}^{4} \cdot \mu_b \cdot \frac{V}{h(t)^3} \qquad (5.4)$$

where μ_b is the Newtonian biaxial elongational viscosity, which is 6 times the Newtonian viscosity in shear.

With Eq. 5.1-5.4, the material constants K, n and μ_b can be calculated from the logF versus logh curve (Fig. 5.2). The slope of the linear part of the logF versus logh curve should equal –n in case of lubricated conditions (see Eq. 5.1) and to –(2n+1) in case of nonlubricated conditions (see Eq. 5.2). For Newtonian materials, the slopes should equal 1 and 3, respectively (see Eq. 5.3 and 5.4). K and Newtonian biaxial viscosity can be calculated from the intercept of the logF versus logh curve.

If the measurement is fully lubricated, the force-height relationship can be recalculated into a stress-strain relationship for pure elongational flow by:

$$\sigma_b = \frac{F_{elongational}}{\pi R_{plate}^2} \quad \text{and} \quad \dot{\varepsilon}_b = \frac{V}{2h(t)} \qquad (5.5,5.6)$$

where σ_b is the biaxial stress, $\dot{\varepsilon}_b$ is the biaxial strain rate and $F_{elongational}$ is the force due to biaxial elongational deformation, that is, the force measured during lubricated squeezing flow. From these data, a flow curve can be constructed for lubricated conditions by plotting the apparent viscosity η against the biaxial strain rate, where $\eta = \sigma_b / \dot{\varepsilon}_b$. For Newtonian materials, the viscosity during biaxial deformation can be calculated for lubricated as well as nonlubricated conditions directly from Eq. 5.3 and 5.4.

Eq. 5.1 to 5.6 are valid for squeezing flow in an ideal set-up and under ideal conditions. In case of imperfect lubricated squeezing flow of foods in a Teflon geometry, a number of factors may affect the measurements and disturb ideal behavior. These factors will be explored in this paper.

Firstly, buoyancy is a factor relevant in imperfect squeezing flow measurements, because the sample is held in a container. The force of buoyancy increases during compression as the upper part of the geometry is further and further immersed in the sample and is given by:

$$B(h) = \frac{(H_{ini} - h(t)) \cdot R^2_{container} \cdot \pi R^2_{plate}}{R^2_{container} - R^2_{plate}} \cdot \rho \cdot g \qquad (5.7)$$

where B(h) is the buoyancy, H_{ini} is the initial sample height at the start of compression, $R_{container}$ is the inner radius of the container, ρ is the density of the product and g is the gravitational acceleration.

Material behavior of the product, such as the presence of yield stress, will affect the behavior of the product during and after compression. The equations given for the relation between the force and sample height during squeezing flow (Eq. 5.1-5.4) can be adjusted for this factor by adding an extra term describing the contribution of yield stress (Engmann *et al.* 2005). Similarly, the measured compression force can be corrected for yield stress and buoyancy such that Eq. 5.1-5.6 still apply:

$$F_{corrected} = F_{measured} - YS \cdot \pi \mathrm{R}_{\mathrm{plate}}{}^2 - B(h) \qquad (5.8)$$

where YS is the apparent yield stress of the material. The yield stress can be obtained from shear measurements (Engmann *et al.* 2005) or from the residual force in the squeezing flow measurement. This latter force, measured after halting the compression movement, can be taken as a measure of the material's yield stress (Suwonsichon & Peleg 1999; Corradini *et al.* 2000a):

$$R = YS \cdot \pi R^2_{plate} + B(1mm) \qquad (5.9)$$

where R is the residual force, consisting after 2 min of relaxation. The yield stress obtained by Eq. 5.9 is, however not the true yield stress, rather a 'recovered' yield stress, because the sample has already been deformed (Corradini *et al.* 2000a).

An alternative way to correct for the presence of yield stress and buoyancy is suggested by Corradini *et al.* (2000a); an 'empirical' flow index is calculated from the ratio of compression forces measured at different compression speeds:

$$\frac{(F_{V_1} - R)}{(F_{V_2} - R)} = \left(\frac{V_1}{V_2}\right)^m \qquad (5.10)$$

where F_{V1} and F_{V2} are forces measured at a standard height during compression at two different compression speeds and m is a constant. The parameter m is considered a material property, independent from the ratio of compression speeds. For Newtonian materials, m equals 1 and for pseudoplastic materials m equals the flow index (Corradini *et al.* 2000a).

Next to yield stress, food products can also exhibit more complex material behavior, such as (pseudo-) thixotropy, viscoelastic behavior, sample inertia, and phase separation of particles or fluid. This will also affect the compression results. Because of their dependency on strain and time, the role of these factors will increase with compression speed.

Another potentially important factor is the presence and extent of friction. The imperfect squeezing flow measurement in a Teflon geometry is intended to operate at lubricated, pure elongational flow conditions. Some studies (Corradini *et al.* 2000a; Corradini *et al.* 2000b) however, show that Teflon cannot fully prevent friction between the sample and the surface of the geometry, resulting in shear flow along with elongational flow. Because of the presence of friction the measured compression force will be much higher (Damrau & Peleg 1997). Just as it is hard to achieve complete slip in lubricated flow, it is also hard to achieve ideal friction in nonlubricated squeezing flow. This can result in an intermediate situation of partial slip (Campanella & Peleg 2002), in which a certain amount of friction is present. The contribution of friction becomes more relevant at higher compression speeds.

A last factor that might affect the measurements and cause deviations from ideal behavior consists of instrumental artifacts. The imperfect set-up of the measurement causes some important artifacts, such as annular flow between the upper part of the geometry and the wall of the container, uneven surface of the product in the container and entry- and end effects. In addition, tilting of the upper part of the geometry and compliance of the machine can play a role (Hoffner *et al.* 2001), especially at small sample heights.

Materials and Methods

Materials

Three commercially available vanilla flavored custards and three commercially available mayonnaises were selected to represent the two types of products. Custard is a typical Dutch dairy dessert product, containing milk, starch, carrageenan and sugar. One custard sample was based on soymilk instead of milk and contained 1.8% fat. Fat content of the other custards are 0.1 and 2.6%. Mayonnaise samples included two full-fat mayonnaises (68 and 80% fat) and one low-fat dressing (31% fat) marketed as mayonnaise-replacer. Both types of products were bought at local supermarkets in The Netherlands and stored at 5°C (custard) or room temperature (mayonnaise). All measurements were performed within a few days after opening the cartons and jars. Extra measurements were performed with arachid oil (Bebo arachid oil, Romi Smilfood BV, Heerenveen, The Netherlands) and treacle syrup (De Originele suikerstroop, CSM Suiker BV, Amsterdam, The Netherlands), both stored at room temperature. Densities of the different types of products have been determined for custards (1070 kg/m^3), arachid oil (900 kg/m^3) and treacle syrup (1419 kg/m^3). For mayonnaises, densities have been estimated at 1000 kg/m^3 for low-fat mayonnaises and 910 kg/m^3 for full-fat mayonnaises (Elert 2005). The actual value for the density is not very important, as its effect on the results is small.

Compression method

Compression measurements were performed with a Texture Analyser (Stable Micro System XT2i) equipped with a 25 kg load cell and Texture Expert Exceed 2.51 software (Stable Micro Systems, Etten-Leur, The Netherlands). The geometry had been manufactured according to Suwonsichon & Peleg (1999). Both parts of the geometry were made out of Teflon, with a diameter of 12 and 14 cm for the plate and container, respectively. A smaller plate of 10 cm was abandoned because reproducibility of the results was lower compared to the larger plate. In case of custards, samples were poured in the container in which a mark at 7 mm height had been drawn. Care was taken to fill the container as close as possible to this mark. In case of the other types of products, a constant amount of product was loaded in the container by spoon or pouring, to ensure a reproducible initial sample height of about 7 mm, that is, 115, 70 and 156 g for the mayonnaises, arachid oil and treacle syrup, respectively. The exact initial height of the sample was determined as the sample height at the moment the compression force exceeded 1 N (custard) or calculated from the sample weight and density (mayonnaises, arachid oil and treacle syrup). In some cases, the uneven surface of the sample was flattened with a spoon, which did not affect the results significantly.

Measurements were performed at room temperature (23°C). Before compression started, samples were allowed to relax and thermally equilibrate for 5 min (custard), 10 min (mayonnaise) or directly measured (arachid oil, treacle syrup). For the lower compression speeds, the waiting time was decreased to compensate for the time required by the upper part of the geometry to reach the surface of the sample. Compression was performed at constant velocity until a sample height of 1 mm was reached. In this position, the upper part of the geometry was held for 2 min before decompression. Data was acquired in the compression and relaxation step, at a rate depending on the compression speed (about 10 measurement points per mm compression). Measurements were made in triplicate, except for the experiment of custards at different initial sample heights.

In the experiments where the initial height was varied, different amounts of sample were loaded into the container, yielding initial sample heights between 5 and 10 mm. The number of different products and compression speed(s) tested varied per product type. For custards, all three products were measured, at 0.1 mm/s. For the mayonnaises, one product

was measured, at 0.25 and 5 mm/s. Arachid oil was measured at 0.25 and 5 mm/s and treacle syrup at 5 mm/s.
For the experiments at different compression speeds, speeds were varied between 0.06 and 10 mm/s. Seven or eight different compression speeds were tested per product, except for arachid oil (0.25 and 5 mm/s). In one experiment with low-fat mayonnaise, lubrication was enhanced by applying arachid oil as a lubricant onto the surface of both parts of the geometry. With a brush 2-3 g of oil was applied, corresponding to a layer of approximately 0.2 mm.

Parameters that were derived from the compression measurements include the stress during compression at 2 mm sample height and at 1mm sample height (equaling the maximum compression force); and the force during the relaxation step at 1 min and at 2 min (see Fig. 5.1 and 5.2). Stresses were obtained by dividing the forces by the surface of the upper part of the geometry. The slope and intercept at h = 1 mm were calculated by fitting the linear part of the logF versus logh curve (see Fig. 5.2). The fitted range covered approximately the last 2 mm of compression for custards and mayonnaises and the last 1 mm of compression for arachid oil and treacle syrup.

Shear measurements

Flow curves in shear have been measured on a Paar Physica MCR 300 rheometer (Anton Paar Benelux, Sint-Martens-Latem, Belgium). For custards, mayonnaises and treacle syrup the rheometer was equipped with a 40 mm plate/plate system (PP40), on which waterproof sandpaper (kor 80) was attached to prevent slippage of the samples. Viscosity of arachid oil has been measured in a concentric cylinder (CC27) system, with the inner cylinder having a diameter of 27 mm and a gap of 1.13 mm. All measurements were performed at room temperature, and waiting times for the sample to relax and thermally equilibrate are similar to those used in the compression measurement. The power-law flow index and Newtonian viscosity were calculated over the range from 0.1 to 100 s^{-1}, except for arachid oil where the range was 1-1000 s^{-1}. More details on the measurements and calculations, especially for custards and mayonnaises, can be found in Chapter 3 and Janssen *et al.* (2007).

Results and Discussion

In general, results of the squeezing flow measurements were very reproducible (see Fig. 5.4-5.8). Errors margins of the extracted parameters (see Table 5.1-5.5), based on the replicates did generally not exceed 15%. Results of mayonnaise with arachid oil as lubricant were the least reproducible (see Fig. 5.7). A linear region, representing predominantly squeezing flow, can be seen in the curves of logF versus logh (Fig. 5.2) and the flow curves of custards and mayonnaises (Fig. 5.4, 5.6 and 5.7). For arachid oil and treacle syrup however, the region of squeezing flow is not linear (see Fig. 5.5 and 5.8). For arachid oil, this might have been caused by turbulence or other undesirable effects due to the low viscosity of the product. In case of treacle syrup, it might have been caused by tilting and/or compliance of the upper part of the geometry and arm of the Texture Analyser (Hoffner *et al.* 1997), as these effects are especially significant for high viscosity products. Ranges of values for the parameters extracted from the compression curve, measured at a range of initial sample heights at one specific compression speed are given in Table 5.1-5.4 for the different types of products. The 'recovered' yield stress, calculated from the stress after 2 min of relaxation by Eq. 5.9 varies between 100 and 180 Pa for the custards and 600 and 880 Pa for the mayonnaises. When parameters obtained at one compression speed and one initial height are compared, they are significantly different (ANOVA, $p < 0.05$) among the different types of products, as well as among the three different custards and mayonnaises. This means that the current squeezing flow method is sensitive enough to distinguish differences between the custard samples and between the mayonnaise samples.

Table 5.1 Ranges of values for the squeezing flow parameters of custards measured at different initial sample heights and one specific compression speed, including the significance of the effect of initial sample height on these parameters.

All custards (V = 0.1 mm/s)	Measured range[a]	Effect of initial sample height[b]		
		no correction	correction for YS	correction for YS+B
Initial sample height (mm)				
custard 0.1% fat	4.7 - 10.0			
custard 2.6% fat	5.0 - 8.2			
custard soy-based	5.9 - 9.1			
Stress at 2 mm compression (kPa)				
custard 0.1% fat	0.7 - 0.9	**	**	ns
custard 2.6% fat	0.5 - 0.7	**	**	ns
custard soy-based	0.8 - 1.0	**	**	ns
Stress at 1 mm compression (kPa)				
custard 0.1% fat	1.3 -1.5	ns	ns	ns
custard 2.6% fat	0.9 - 1.3	*	*	ns
custard soy-based	1.3 -1.6	ns	ns	ns
Stress at 1 min relaxation (kPa)				
custard 0.1% fat	0.3 - 0.5	**	**	ns
custard 2.6% fat	0.3 - 0.4	**	**	ns
custard soy-based	0.3 - 0.5	**	**	ns
Stress at 2 min relaxation (kPa)				
custard 0.1% fat	0.3 - 0.5	**	**	-
custard 2.6% fat	0.3 - 0.4	**	**	-
custard soy-based	0.3 - 0.5	**	**	-
Slope from logF vs. logh				
custard 0.1% fat	-0.7 - -0.9	*	**	ns
custard 2.6% fat	-0.8 - -1.2	**	**	ns
custard soy-based	-0.7 - -0.9	**	*	ns

a The error margins of the values (calculated from the replicates) are less than 15% for all extracted parameters.

b Significance of effect tested by linear regression: ns not significant for $p < 0.05$; * significant for $p < 0.05$; ** significant for $p < 0.01$.

Flow-curve measurements in shear on arachid oil and treacle syrup show that these products behave as Newtonian materials in the measured range (see Fig. 5.5 and 5.8). Newtonian viscosities over this shear rate range are 0.07 Pas and 18 Pas for arachid oil and treacle syrup, respectively.

Table 5.2 Ranges of values for the squeezing flow parameters of mayonnaises measured at different initial sample heights and one specific compression speed, including the significance of the effect of initial sample height on these parameters.

Mayonnaise, 80% fat (V = 0.25 and 5 mm/s)	Measured range[a]	Effect of initial sample height[b]		
		no correction	correction for YS	correction for YS+B
Initial sample height (mm)				
V = 0.25 mm/s	5.4 - 10.7			
V = 5 mm/s	5.4 - 10.7			
Stress at 2 mm compression (kPa)				
V = 0.25 mm/s	3.5 - 4.0	ns	*	ns
V = 5 mm/s	7.0 - 8.5	ns	ns	ns
Stress at 1 mm compression (kPa)				
V = 0.25 mm/s	5.8 - 6.5	ns	ns	ns
V = 5 mm/s	11.3 -13.3	ns	ns	ns
Stress at 1 min relaxation (kPa)				
V = 0.25 mm/s	1.0 - 1.2	ns	**	ns
V = 5 mm/s	1.0 - 1.5	ns	*	ns
Stress at 2 min relaxation (kPa)				
V = 0.25 mm/s	0.9 -1.0	*	**	-
V = 5 mm/s	0.9 - 1.2	*	**	-
Slope from logF vs. logh				
V = 0.25 mm/s	-0.75 - -0.89	**	**	**
V = 5 mm/s	-0.77 - -0.88	**	**	**
Intercept from logF vs. logh				
V = 0.25 mm/s	0.2 - 0.4	**	**	**
V = 5 mm/s	0.2 - 0.8	**	**	**

a The error margins of the values (calculated from the replicates) are up to 50% for the intercept from the logF vs. logh curve and less than 15% for the other extracted parameters.

b Significance of effect tested by linear regression: ns not significant for $p < 0.05$; * significant for $p < 0.05$; ** significant for $p < 0.01$.

Effect of initial sample height

Compression measurements with different initial sample heights (5-10 mm) at low and/or high compression speeds were performed with three custard products, one of the mayonnaises, arachid oil and treacle syrup. Many of the extracted parameters, for example the slope, stress at 2 mm, stress at 1 min and at 2 min relaxation time are significantly affected by initial height for most product types and for both compression speeds (see Table 5.1-5.4). The values of the parameters increase with increasing initial sample height. An example for the stress at 2mm of one of the custards is shown in Fig. 5.3. The effect of initial sample height is not significant for parameters representing high force values, that is, stress at 1 mm of compression for highly viscous products such as treacle syrup and mayonnaises (see Table 5.1-5.4). The effects of initial sample height persist after the parameters of custards and mayonnaises have been corrected for the presence of yield stress by Eq. 5.8, using the 'recovered' yield stress of Eq. 5.9 (see Table 5.1 and 2 and Fig. 5.3).

Table 5.3 Ranges of values for the squeezing flow parameters of arachid oil measured at different initial sample heights and one specific compression speed, including the significance of the effect of initial sample height on these parameters.

Arachid oil (V = 0.25 and 5 mm/s)	Measured range[a]	Effect of initial sample height[b]	
		no correction	correction for B
Initial sample height (mm)			
V = 0.25 mm/s	4.7 - 8.8		
V = 5 mm/s	4.7 - 15.2		
Stress at 2 mm compression (kPa)			
V = 0.25 mm/s	0.097 - 0.225	**	**
V = 5 mm/s	0.27 - 0.45	**	ns
Stress at 1 mm compression (kPa)			
V = 0.25 mm/s	0.154 - 0.288	**	ns
V = 5 mm/s	0.81 - 0.97	ns	ns
Stress at 1 min relaxation (kPa)			
V = 0.25 mm/s	0.12 - 0.26	**	ns
V = 5 mm/s	0.12 - 0.27	**	ns
Stress at 2 min relaxation (kPa)			
V = 0.25 mm/s	0.12 - 0.26	**	ns
V = 5 mm/s	0.12 - 0.27	**	ns
Slope from logF vs. logh			
V = 0.25 mm/s	-0.37 - -0.79	**	*
V = 5 mm/s	-1.3 - -1.8	**	ns
Intercept from logF vs. logh			
V = 0.25 mm/s	0.1 - 0.3	**	ns
V = 5 mm/s	$0.5 \cdot 10^5$ - $140 \cdot 10^5$	**	ns

a The error margins of the values (calculated from the replicates) are up to 75% for the intercept from the logF vs. logh curve and less than 15% for the other extracted parameters.

b Significance of effect tested by ANOVA: ns not significant for $p < 0.05$; * significant for $p < 0.05$; ** significant for $p < 0.01$.

The effect of initial sample height is in disagreement with the theory of ideal squeezing flow. Hoffner *et al.* (1997) reported no effect of initial height in their results on imperfect squeezing flow, but the variation in initial sample height was less broad in their experiment and their products were relatively higher in viscosity. The observations on the effect of initial sample height can be attributed to the imperfect nature of the method used, causing buoyancy effects and instrumental artifacts, such as annular flow between the upper part of the geometry and the wall of the container. Buoyancy force is affected by the initial sample height, because the immersion depth at a specific compressed height becomes larger with increasing sample volume (see also Eq. 5.7). The contribution of buoyancy to the measured stress is limited to 0.1 to 0.4 kPa for the different types of products at maximum compression. Instrumental artifacts such as entry- and end effects and the uneven surface of the product in the container are also caused by the imperfect set-up, but they are less likely to be responsible for the effect of initial sample height. These three issues are especially prominent during the initial phase of the compression, where logF versus logh is not yet linear, and can be neglected in the rest of the measurement (Hoffner *et al.* 1997; Campanella & Peleg 2002). The stress involved with annular flow increases with the viscosity of the product, making this instrumental artifact especially relevant for thicker product types (Campanella & Peleg 2002).

Table 5.4 Ranges of values for the squeezing flow parameters of treacle syrup measured at different initial sample heights and one specific compression speed, including the significance of the effect of initial sample height on these parameters.

Treacle syrup (V = 5 mm/s)	Measured range[a]	Effect of initial sample height[b]	
		no correction	correction for B
Initial sample height (mm)	5.0 - 8.9		
Stress at 2 mm compression (kPa)	9 - 15	*	*
Stress at 1 mm compression (kPa)	18 - 25	ns	ns
Stress at 1 min relaxation (kPa)	0.19 - 0.40	**	ns
Stress at 2 min relaxation (kPa)	0.19 - 0.40	**	ns
Slope from logF vs. logh	-0.75 - -1.09	**	**
Intercept from logF vs. logh	0.1 - 1.7	*	*

a The error margins of the values (calculated from the replicates) are up to 75% for the intercept from the logF vs. logh curve, 40% for the stress at 2 and 1 mm and less than 15% for the other extracted parameters.

b Significance of effect tested by linear regression: ns not significant for $p < 0.05$; * significant for $p < 0.05$; ** significant for $p < 0.01$.

So, for low-viscosity products, that is, custards and arachid oil, instrumental artifacts play a minor role, and the effect of initial sample height can be explained solely by buoyancy. Previously significant effects of initial height on most of the parameters become insignificant after correction for buoyancy (and yield stress), see Table 5.1 and 5.3 and Fig. 5.3. The same is true for mayonnaises and treacle syrup regarding the parameters during relaxation (stress at 1 and at 2 min), see Table 5.2 and 5.4. As the effect of instrumental artifacts is negligible during the relaxation step, only buoyancy plays a role. But for the parameters during the compression step (stress at 2 mm, slope, intercept) of these high-viscosity products, the contribution of instrumental artifacts becomes prominent, and correction of buoyancy was not enough to explain the significant effect of initial height on these parameters (see Table 5.2 and 5.4).

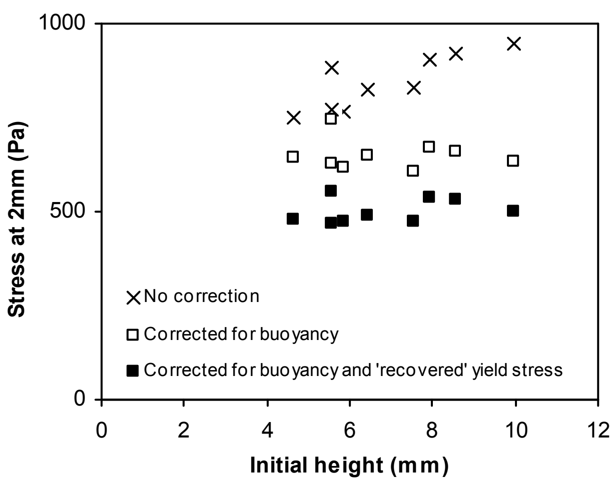

Figure 5.3 Stress at 2 mm compression of soy-based custard without and with correction for buoyancy and yield stress.

Finally, for parameters of mayonnaises and treacle syrup representing high stress values (that is, the stress at 1 mm compression), both contributions of buoyancy and instrumental artifacts are negligible compared to the high compression force, so there is no effect of initial sample height at all on these parameters (see Table 5.2 and 5.4).

The presence of friction instead of slip seems to play no role in the effect of initial sample height on the extracted parameters. If this were the case, one would expect to see different results between measurements at low and high compression speeds. There was however no clear difference in significance between parameters at different compression speeds, see Table 5.2 and 5.3.

Effect of compression speed

Compression speed has been varied at similar sample heights for all three custards and mayonnaises and for arachid oil and treacle syrup. As expected, stresses measured during compression (stress at 1 mm and at 2 mm) increase with increasing compression speed, while stresses during the relaxation step (stress at 1 min and at 2 min) are less affected (results not shown). Table 5.5 shows the range of slopes calculated from the linear part of the logF versus logh curves at different compression speeds and the effect of compression speed on these slopes. The values of the slopes can be calculated into flow indices after establishing whether the conditions are lubricated or nonlubricated (see Table 5.5 and Eq. 5.1-5.4). On the assumption that the measurements are fully lubricated, the force versus height relations can be transformed into a relationship between elongational viscosity and biaxial strain rate by Eq. 5.5 and 5.6. This results in a composed flow curve for each product (Fig. 5.4, 5.5, 5.6, 5.7, 5.8 and 5.9), made up of several smaller flow curves, one for each compression measurement at a specific compression speed, including replicates (see Fig. 5.4). The increase in biaxial strain rate on the x-axis is caused by the decrease in sample height (during a compression measurement at constant speed), and by the change in compression speed (for consecutive measurements). For this reason, measurements at different, increasing compression speeds are located in ascending order on the x-axis (see Fig. 5.4).

The values for the slope of the logF versus logh curve (see Table 5.5) and the Newtonian elongational viscosities (see Fig. 5.5 and 5.8) are different from those expected for lubricated conditions. In case of the Newtonian products, arachid oil and treacle syrup, biaxial elongational viscosity should be six times the viscosity in shear and should be independent from strain rate. Hence, flow curves of these products, assuming lubricated conditions, should lie horizontal at a height of six times the viscosity in shear. As the flow index should be 1.0, the slope of the logF versus logh curves should be -1.0 for lubricated flow (see Eq. 5.3 and Table 5.5). None of these expectations are met in the results for both Newtonian products; both flow curves are not horizontal, viscosities are much higher than those predicted from viscosities in shear (see Fig. 5.5 and 5.8), and the slopes of logF versus logh are not close to -1.0 (see Table 5.5). For the non-Newtonian products, the custards and mayonnaises, viscosity should be strain-dependent. Measurements on the flow index in shear showed strain-rate thinning behavior with flow indices of 0.1 to 0.2 for both products (see Table 5.5 and Chapter 3). Therefore, we assume similar behavior in elongation, with flow indices smaller than 1.0, and slopes of the logF versus logh curve between 0 and -1.0 (lubricated flow, see Eq. 5.1 and Table 5.5). The values measured in elongation for the custards and mayonnaises in this study do show strain-rate thinning behavior with slopes just below or close to -1.0, but these slopes are much higher than expected based on shear measurements. According to Campanella & Peleg (2002), values around 1.0 indicate that the conditions for lubricated squeezing flow have not been met.

Table 5.5 Ranges of slopes measured at different compression speeds, flow indices at constant strain and in shear and ranges of values for m for the different types of products. For the slopes, the significance of the effect of compression speed is included. The flow indices at 0.75 strain are calculated from a power law fit through the data points at 0.75 strain. The m-values are given as a range because calculations have been performed for all possible combinations of V_1 and V_2 in Eq. 5.10; the significance of the effect of the average of V_1 and V_2 is included.

	Range of V (mm/s)	Slope from logF vs. logh[a] lubr: flow index = -slope (see Eq. 5.1) nonlubr: flow index = -½(slope+1) Eq. 5.2		Flow index at constant strain		m		In shear
		measured range	effect of V[b]	flow index	R^2	calculated range	effect of average V1,V2[b]	flow index
Custard								
custard 0.1% fat	0.05-10	-0.68 - -0.94	*	0.25	0.999	0.21 - 0.78	ns	0.13
custard 2.6% fat	0.05-10	-0.99 - -1.17	*	0.29	0.994	0.06 - 0.66	ns	0.22
custard soy-based	0.05-10	-0.86 - -1.05	**	0.30	0.996	0.10 - 1.05	ns	0.20
Mayonnaise								
mayonnaise 31% fat	0.06-10	-0.59 - -0.86	ns	0.34	0.997	0.27 - 0.61	**	0.22
mayonnaise 68% fat	0.08-10	-0.82 - -0.89	ns	0.25	0.999	0.25 - 0.41	ns	0.09
mayonnaise 80% fat	0.08-10	-0.77 - -0.83	ns	0.22	0.999	0.10 - 0.41	ns	0.14
With lubricant								
mayonnaise 31% fat	0.08-10			0.44	0.992	0.34 - 0.75	ns	0.22
Arachid oil	0.25, 5	-0.79 - -1.81	**			1.02 - 1.07	-	1.00
Treacle syrup	0.08-10	-0.72 - -1.58	**		0.898	0.40 - 0.69	**	0.99

a The error margins of the values (calculated from the replicates) are less than 15%.

b Significance of effect tested by linear regression and by ANOVA (for arachid oil): ns not significant for $p < 0.05$; * significant for $p < 0.05$; ** significant for $p < 0.01$.

Another observation that disagrees with the theory of ideal lubricated squeezing flow is the effect of compression speed on the results. Elongational properties such as the flow index should be independent from measurement conditions such as compression speed. So the slopes of the logF versus logh curve should be similar for different speeds. Elongational viscosity, although rate-dependent should also not be affected directly by compression speed (Corradini *et al.* 2000a). For this reason, the flow curves for the different compression speeds within a composed flow curve should be aligned (see Corradini *et al.* 2000a; Campanella & Peleg 2002), having similar slopes (reflecting flow indices) and intercepts (reflecting consistencies). In our results, however, there is an effect of compression speed. The slopes of the logF versus logh curve are significantly affected by compression speed for all products except mayonnaises (see Table 5.5), and the composed flow curves did not align for any of the products (see Fig. 5.4-5.9).

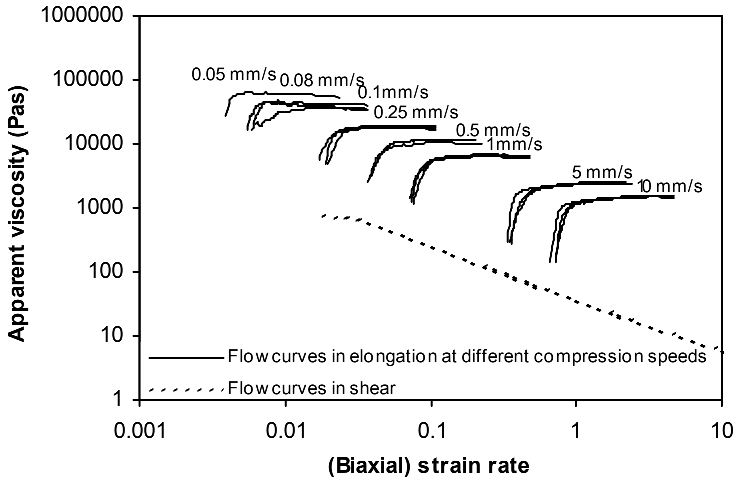

Figure 5.4 (Composed) Flow curve of soy-based custard in elongation and in shear; the composed flow curve in elongation is composed of multiple curves measured at different compression speeds including replicates. The compression speed per set of replicate curves is indicated in the graph.

So the results obtained by imperfect squeezing flow in a Teflon geometry at different compression speeds are different than those expected for theoretical, ideal lubricated squeezing flow. In the following, several factors that could be responsible for the deviating behavior of slopes and flow curves, and the effect of compression speed on it are considered one by one. One such factor is the yield stress behavior of custards and mayonnaises, which was not included in the results of Table 5.5 and Fig. 5.4-5.9 so far. Another factor is buoyancy, which is due to the imperfect nature of the geometry. Although both factors are relevant, and buoyancy appeared to be a main factor in the effect of initial sample height, they are of limited influence when considering the effect of compression speed on the slope of the logF versus logh curve and the flow curve. This is shown in Fig. 5.6, where the flow curves of full-fat mayonnaise have been corrected by the 'recovered' yield stress and buoyancy according to Eq. 5.7, 5.8 and 5.9. The elongational viscosity does decrease slightly, but the flow curves per measurement become even less well aligned than without correction. The slopes of the logF versus logh curve are slightly increased for most products when corrected, but they are still different from expected values (results not shown).

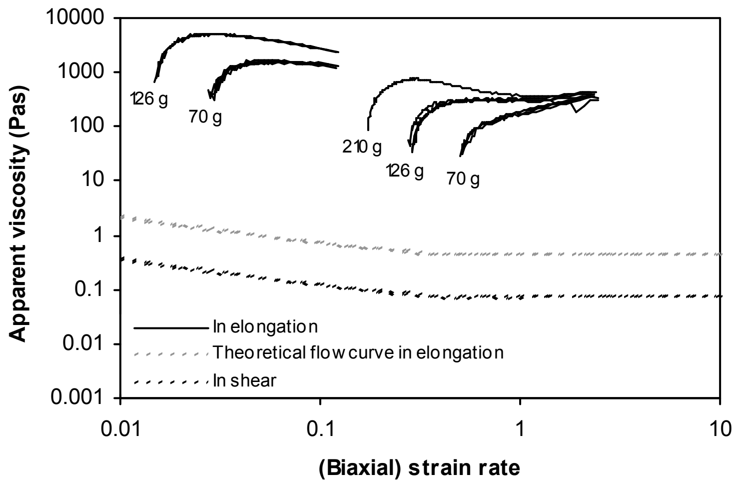

Figure 5.5 (Composed) Flow curve of arachid oil in elongation and shear, as well as the theoretical elongation viscosity predicted from the viscosity in shear.

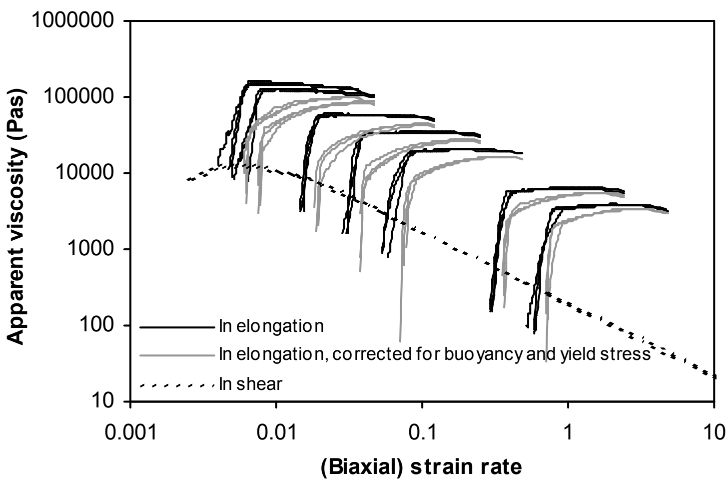

Figure 5.6 (Composed) Flow curves of full-fat mayonnaise (68% fat) without and with correction for 'recovered' yield stress and buoyancy.

A plausible explanation for the discrepancies found is that the requirement of full lubrication has not been met in our measurements. From the types of food tested in this study, mayonnaises are most likely to experience slip, because of its self-lubricating character due to pseudoplasticity and the potential exudation of oil from the product creating an oily slip layer. For this reason, a mayonnaise was chosen to investigate the presence of lubricated conditions. In this experiment, lubrication was further enhanced by applying a layer of arachid oil to the surface of both parts of the geometry before compression of low-fat mayonnaise. Arachid oil acts in this case as a lubricant, because its viscosity is much lower than that of mayonnaise. Figure 5.7 shows that viscosity decreases when a lubricant is applied, indicating that slip was not complete in the compression without lubricant. So, the condition of full lubrication seems not valid for the measurements in this study, even at low compression speeds.

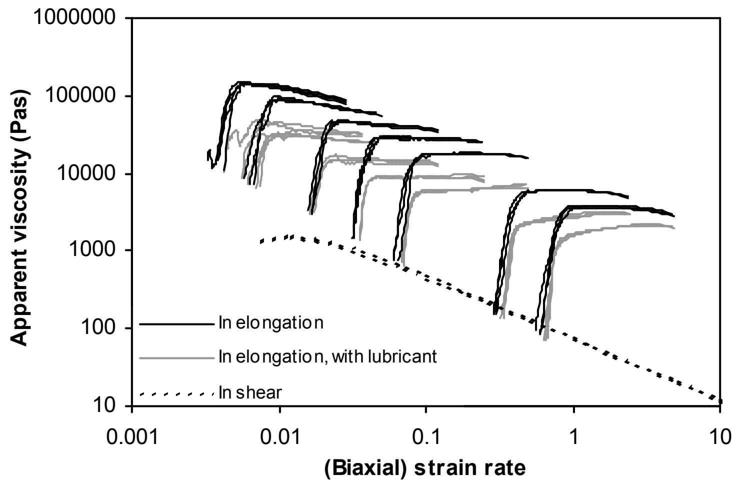

Figure 5.7 (Composed) Flow curves of low-fat mayonnaise without and with arachid oil as lubricant.

On the other hand, the assumption of nonlubricated conditions, with full frictional flow is also not true for our measurements. When the slopes of the logF versus logh curves (Table 5.5) are calculated into flow indices assuming nonlubricated flow (see Eq. 5.2 and Table 5.5), values close to zero are obtained for the Newtonian products and negative values for most of the custards and mayonnaises. These values seem unlikely from a theoretical point of view. Furthermore, plotting the viscosity of treacle syrup for nonlubricated conditions (Eq. 5.4) in the composed flow curve of Fig. 5.8 does not give a satisfying result either. Although the viscosity has been lowered to the value based on theory, the curves of the separate measurements are still not independent from strain rate and not aligned. As neither full slip nor full frictional conditions can describe the results in this study properly, we assume that the conditions are somewhere in between, that is, partial slip. This condition seems to apply for all compression speeds tested, as results even for the lowest compression speed calculated by lubricated theory, as well as results for the highest speed calculated by nonlubricated theory are not realistic. Some experimental models to determine the extent of friction during partial-slip measurements have been presented by Damrau & Peleg (1997) and Engmann *et al.* (2005), but as they are rather complicated to apply they fall beyond the scope of this work.

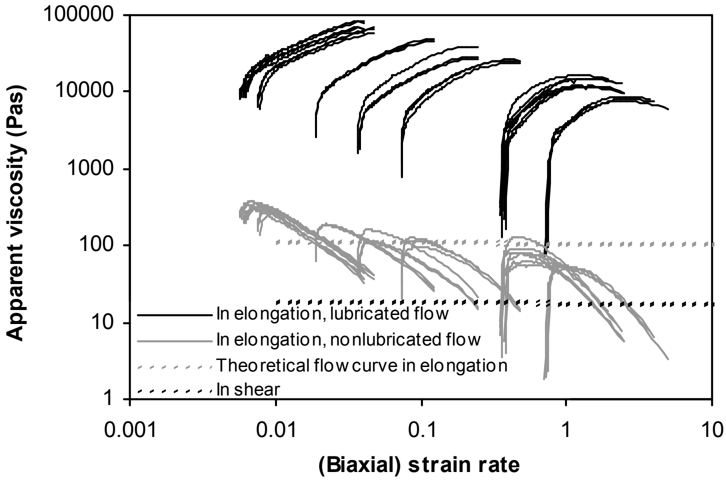

Figure 5.8 (Composed) Flow curves of treacle syrup calculated for lubricated and nonlubricated elongational conditions and in shear, as well as the theoretical elongational viscosity predicted from the viscosity in shear.

The presence of a certain amount of friction cannot solely explain the results of our measurements, at least not for mayonnaises. This is indicated by Fig. 5.7, where the flow curves of mayonnaise are still not aligned after eliminating friction by the use of a lubricant. Other factors involved could be originating in the behavior of the measured products, such as (pseudo-) thixotropy and viscoelasticity. These two factors are known to play a role for custards and mayonnaises (Janssen *et al.* 2007; see Chapter 3). For (pseudo-) thixotropic materials, viscosity decreases during deformation as a result from structure changes. These changes are fully reversible for truly thixotropic materials, otherwise they are pseudo-thixotropic. The (pseudo-) thixotropic behavior of custards and mayonnaises makes the compression measurements sensitive to the applied strain. This can be overcome by comparing results from different compression speeds at the same strain, see Fig. 5.9 for one of the custards. When apparent viscosities of each separate measurement curve are taken at a strain of 0.75, the data points are well aligned and the flow index calculated from its slope is in the same range as the one measured in shear (see Table 5.5). Applying the same approach on arachid oil and treacle syrup is not effective, as both products are not thixotropic. So, in case of custards and mayonnaises, correcting for (pseudo-) thixotropic behavior appears to be successful in obtaining realistic flow indices that are valid over a range of applied compression speeds. The constant-strain approach seems to enable the extraction of elongational properties from squeezing flow measurements. The results obtained should be regarded with care, because other factors, such as the effect of friction, are not included. Also, it is hard to determine the error margin of the calculated flow indices.

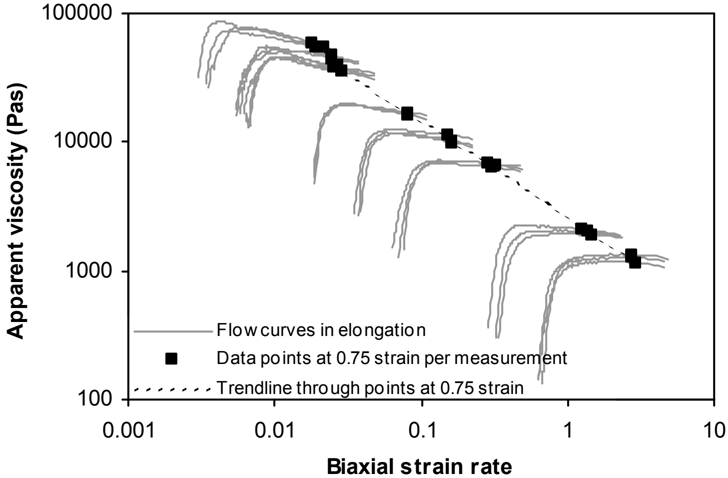

Figure 5.9 (Composed) Flow curve of custard (0.1% fat) with data points at constant strains of 0.75.

(Visco)elastic behavior of custards and mayonnaises could also play a role in the squeezing flow measurements. Elastic properties will only affect the measurement when the relaxation times of the material are comparable or larger than times of squeezing deformation. This might be the case, especially at the higher compression speeds. Other factors explaining the effect of compression speed could be instrumental artifacts (annular flow), especially for thick products such as mayonnaises and treacle syrup. Slight tilting of the upper part of the geometry and compliance of the machine could also have caused deviations in the results (Hoffner *et al.* 2001).

Corradini *et al.* (2000a) suggested the calculation of m (see Eq. 5.10) as an alternative for determining the flow index. m is calculated from the ratio of the compression force measured at two different compression speeds, with the compression force corrected for the force after relaxation. Results of m for the products in this study are inconsistent, see Table 5.5. For arachid oil, m is 1, equal to the expected flow index, but this is not the case for treacle syrup. For custards and mayonnaises, the values of m calculated for different combinations of compression speeds range very broad, especially for the custards. The lower part of this range represents m-values that are close to expected flow indices. In total, it seems that the use of m is not capable of overcoming all the problems encountered in the imperfect squeezing flow measurement of our products. This can be explained by the fact that m only compensates for buoyancy and yield stress, and not for other factors, such as friction and thixotropy. As the extent of friction depends on the compression speed, different extents of friction are discounted in the ratios of Eq. 5.10. This can explain the significant effect of the averaged compression speed per combination of V_1 and V_2 from Eq. 5.10 on m for some of the products (see Table 5.5).

Conclusion

The imperfect squeezing flow technique in a Teflon geometry is an easy method for performing routine measurements on semisolid food products such as custards and mayonnaises to study elongational deformation in relation to oral texture perception. However, the results in this study for custards and mayonnaises do not show behavior predicted by the theories of lubricated or nonlubricated squeezing flow. An effect of initial sample height and of compression speed is shown. Also, calculated values for the flow index are different from expected. The same is true when Newtonian samples were measured. This deviating behavior can be explained by a combination of factors, which have been explored individually. An important factor is the presence of a certain amount of friction, making the application of lubricated theory as well as nonlubricated theory invalid. The condition of partial slip is very important for the effect of compression speed, because of frictional effects that depend on velocity. However, correcting the results for this factor falls beyond the scope of this paper. Next to friction, (pseudo-) thixotropic behavior is important for the results of custards and mayonnaises at different compression speeds, but as expected, not for the results of both Newtonian products. Correcting for the strain history of custards and mayonnaises appears to be a successful way of obtaining a realistic flow index, although the results should be regarded with care. The presence of buoyancy during the measurement does affect the results, especially for low viscous products and parameters representing low stress values. The effect of initial sample height for arachid oil and custards can be explained by the presence of buoyancy. Yield stress behavior is observed for custards and mayonnaises and therefore should be included in the calculations, but cannot explain the deviating results. The calculation of m as an alternative material property for the flow index was not successful in overcoming the effect of compression speed, as this calculation only corrects for buoyancy and yield stress. Other factors that seem to play a role are more difficult to quantify, such as the elastic response of the products and instrumental artifacts. The latter are especially important for treacle syrup and can explain the results of the effect of initial sample height for the more viscous products. The effect of instrumental artifacts can be further minimized by adapting the design of the geometry. As quantitatively correcting for all of the factors mentioned above is not possible at this point, it becomes difficult to deduce pure elongational properties from the measurements. Imperfect squeezing flow in a Teflon geometry is a very practical technique to measure semisolids such as custards and mayonnaises under (partly) elongational deformation, but results should be regarded more qualitative than quantitative.

Acknowledgments

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6

SQUEEZING FLOW MEASUREMENTS OF SEMISOLID FOODS AND RELATIONS WITH TEXTURE PERCEPTION

Abstract

The technique of imperfect squeezing flow (Corradini *et al.* 2000a), measuring under conditions of (partial) elongational flow was applied to characterize a set of commercial mayonnaises and a set of commercial custards. Values of parameters extracted and calculated from the compression, relaxation and decompression step were compared to properties measured in shear and were related to sensory attributes in a univariate and multivariate way. Results showed that the squeezing flow method is an easy and practical method that is sensitive enough to distinguish differences within the set of custard and mayonnaise samples. For custards, squeezing flow properties were mainly related to creaminess and fattiness. Other attributes were poorly predicted due to the lack of saliva-induced breakdown in the measurement. Custards were perceived as creamier when the product showed a steep increase in stress during compression and limited stress decay after halting the compression. For mayonnaises, attributes were strongly related to parameters reflecting density and fat content rather than rheological properties. This was partly due to the limited differences in thickness of the mayonnaises under study. For custards, imperfect squeezing flow measurements seem useful next to shear measurements, especially for the attribute creaminess, and elongational deformation seems relevant for oral processing and perception. For the mayonnaises in this study, this is less true, given the currently applied measurement technique. Investigations on the meaning and validity of the material properties measured in the imperfect squeezing flow measurement showed that the 'recovered' yield stress from the squeezing flow measurement reflects a different material property than the conventional yield stress. From the three different ways to obtain a flow index from the squeezing flow measurement, only the constant-strain approach and the alternative flow index approach may lead to a valid flow index in elongation. The imperfect squeezing flow technique provides practical, but primarily qualitative measurements that are especially applicable in relation to the perception of texture of custards.

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Introduction

Perceived texture is an important factor in the judgment of food quality by consumers. For a large number of semisolid food products, quality is positively associated with the attribute creaminess. To optimize the quality of food products, it is essential to understand the mechanisms behind perception of creaminess and related attributes. This can be achieved by identifying relations between perceived texture attributes and physicochemical product properties measured under conditions resembling those in the mouth. An extra benefit from this approach is the potential development of instrumental measurements predicting texture attributes as perceived by consumers.

Many authors have reported relations between texture attributes and rheological product properties for semisolids (Wood 1974; Kokini *et al.* 1977; Daget *et al*. 1987; Stanley & Taylor 1993; Wendin *et al.* 1997; Kilcast & Clegg 2002; see Chapter 3 and 4). These studies have focused on rheological parameters measured under shear conditions. However, in the mouth elongational deformation is also present (van Vliet 2002). For this reason, rheological behavior in elongation should also be taken into consideration when identifying relations with orally perceived sensory attributes. The technique of imperfect (lubricated) squeezing flow (Suwonsichon & Peleg 1999) is the only practically available method to measure elongational properties of semisolid products in a way resembling oral conditions. In this method, biaxial elongational flow is established by compression at constant velocity between a plate and a shallow container. Extra information is obtained from the stress relaxation step after compression (Corradini *et al*. 2000a) and from the decompression step, in which the plate is pulled up again. Previous research (Corradini *et al.* 2000a; Campanella & Peleg 2002) has shown that despite the use of Teflon, the requirement of full lubrication is not always met, implying that conditions during compression involve a combination of elongation and shear.

In Chapter 5 we have explored the imperfect squeezing flow technique for mayonnaises and custards. Results showed the presence of a certain extent of shear next to elongational deformation, as well as the important role of (pseudo-) thixotropic behavior of the products. Also the effect of yield stress, buoyancy and instrumental artifacts on the measurement results was shown to be significant. Because it is not possible to correct for all of these factors, the squeezing flow measurement should be considered more qualitative than quantitative (see Chapter 5). In the current study, the imperfect squeezing flow technique is applied to characterize a set of commercial mayonnaises and a set of commercial custards. As the sample sets are larger than those used in the previous study, it is possible to further investigate the squeezing flow properties obtained from the measurements and to relate them to orally perceived sensory attributes. The measurements in this study are performed at two different compression speeds; at low speed (0.25 mm/s) to minimize the contribution of shear, and at high speed (5 mm/s) to simulate deformation rates present during oral processing (Prinz, personal communications). The aim of the study was, first, to get more insight in the meaning and validity of material properties obtained from squeezing flow (i.e., yield stress and flow index), by comparing them to the same properties obtained from shear measurements. Furthermore, relations were established between sensory attributes and parameters determined in the imperfect squeezing flow measurement for mayonnaises and custards. Comparing these relations to the relations found with parameters measured in shear gives insight in the applicability of measurements under (predominantly) elongational conditions in comparison to shear measurements as well as in the role of elongational flow in oral processing and perception.

Materials and Methods

Materials

A set of fourteen commercially available mayonnaises and a set of fourteen commercially available vanilla flavored custards were used for this study. The mayonnaise samples included a group of full-fat mayonnaises (67-80% fat) and a group of low-fat dressings (11-41% fat) marketed as mayonnaise-replacer. As the variation in thickness of the mayonnaise samples was not large, the differences in fat content played a dominant role in this sample set (see Chapter 3). Custard is a typical Dutch dairy dessert product, containing milk, starch, carrageenan and sugar. Two custard samples were based on soymilk instead of milk. Fat content of the custards ranged between 0.1 and 3.5%. Both types of products were bought at local supermarkets in The Netherlands and stored at 23°C (mayonnaises) or 5°C (custards) prior to testing. All measurements were performed within a few days after opening the cartons and jars.

Squeezing flow measurements

Imperfect (lubricated) squeezing flow measurements were performed with a Texture Analyser (Stable Micro System TA-XT2i) equipped with a 25 kg load cell and Texture Expert Exceed 2.51 software (Stable Micro Systems, Etten-Leur, The Netherlands). Upper and lower geometries have been manufactured according to Suwonsichon & Peleg (1999), with a diameter of 12 and 14 cm for the probe and container, respectively, and both made out of Teflon. In case of custards, samples were taken directly from the refrigerator and poured in the container in which a mark at 7 mm height had been drawn. Care was taken to fill the container as close as possible to this mark to ensure a uniform initial sample height of about 7 mm. In case of mayonnaises it was more practical to load the container with a constant weight of product (100 g), by spoon or pouring. In some cases, the uneven surface of the sample was flattened with a spoon, but this did not affect the results significantly.

All measurements were performed at room temperature. Before the compression started, samples were allowed to relax and thermally equilibrate for 5 min (custards) or 10 min (mayonnaises). In case of the low compression speeds, the waiting time was decreased to compensate for the time required by the probe to reach the surface of the sample. Compression was performed at a constant velocity of 0.25 or 5 mm/s until a sample height of 1 mm was reached. In this position, the probe was held for 2 min before decompression (Corradini *et al*. 2000a). See Fig. 6.1a for an example of a measurement curve. Data was acquired in the compression step, holding step and, for mayonnaises, the decompression step, at a rate of 2 (for compression at 0.25 mm/s) or 50 (for compression at 5 mm/s) points per s. All measurements were done at least in triplicate.

Most of the rheological parameters (see Fig. 6.1 and Table 6.1) were extracted or calculated from the compression step of the measurement (see also Chapter 5). These included the stresses at different sample heights and the sample height at which the stress exceeded 100 Pa, at the beginning of the measurement. After plotting the data from the compression step in a logarithmic plot of stress versus height (see Fig. 6.1b), the slope and intercept could be calculated. This slope is, in theory, similar to the flow index. From the holding step, the stress after 2 min of relaxation was calculated into the 'recovered' yield stress (Corradini *et al*. 2000a) by compensating for buoyancy (see Chapter 5), see Fig. 6.1a and Table 6.1. For mayonnaises, also the energies involved in compression and decompression were determined, as well as the peak stress during decompression and the mass of product sticking to the upper geometry after the measurement (see Fig. 6.1a and Table 6.1). All these parameters were extracted or calculated for measurements at low (l) and high (h) compression speed. In addition, most of the parameters had alternative versions corrected for buoyancy and yield stress (see Chapter 5). Furthermore, by combining results from measurements at both compression speeds, an alternative flow index m and thixotropy-corrected flow index n0.75 were calculated (see Table 6.1 and Chapter 5). The initial height required for the calculation of buoyancy forces was calculated from the sample weight and density (for mayonnaises) or assumed as the sample height when the compression force exceeded 100 Pa (for custards). The density of the products was estimated for mayonnaises (1000 kg/m^3; Elert 2005) and determined by measurement for custards (1070 kg/m^3). The actual value for the density is not very important, as its effect on the buoyancy results is small.

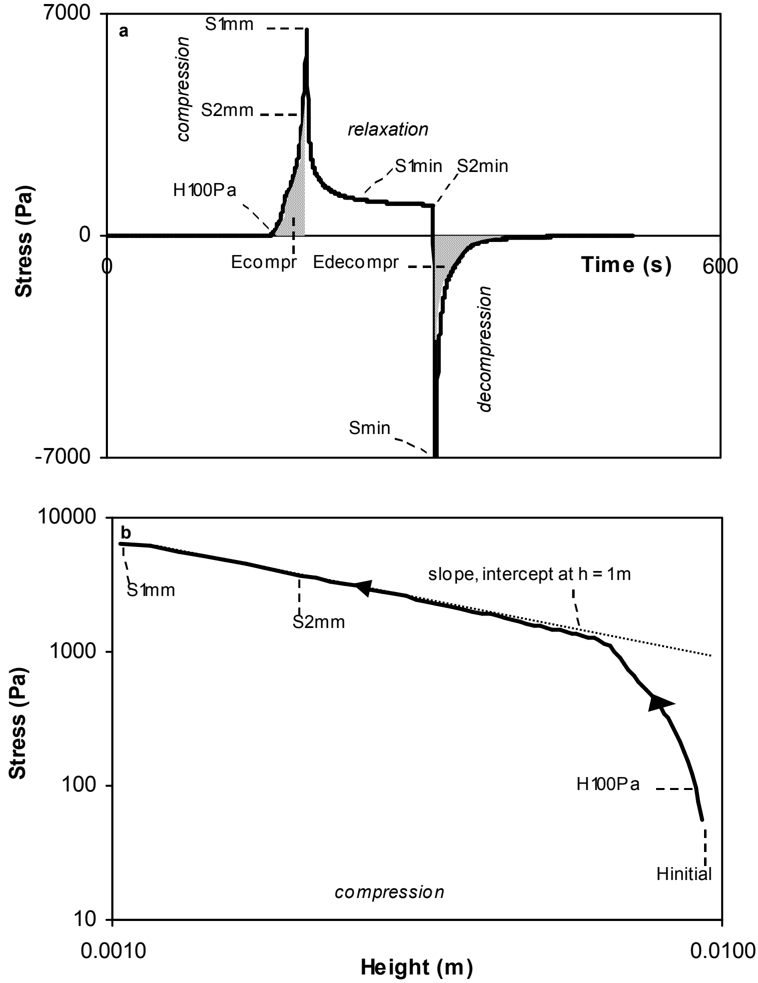

Figure 6.1a,b Example of a curve obtained by the squeezing flow measurement, including the parameters that are directly extracted and calculated from the curve. (a) shows the complete squeezing flow measurement, (b) only the compression step, on logarithmic axes.

Table 6.1 Rheological parameters extracted and calculated from the squeezing flow measurement, including the ranges measured for custards and mayonnaises.

	Code*	Parameter	Determination	Measured range* Mayonnaise	Measured range* Custard
Compression	H100Pa	**Sample height at 100 Pa (mm)**	Height of the sample when the compression force exceeds 100 Pa	l: 6.41-7.82 h: 6.93-8.00	l: 7.7-9.75 h: 7.78-10.20
	S2mm	**Stress at 2 mm (Pa)**	Extracted from compression data	l: 2438-4023 h: 6148-8429	l: 744-1514 h: 1564-3959
	S2mmBY	corrected for B and YS	idem, with force corrected for buoyancy at 2 mm and 'recovered' yield stress	l: 1212-2747 h: 5010-7087	l: 371-1063 h: 1212-3494
	S1mm	**Stress at 1 mm (Pa)**	Extracted from compression data	l: 4232-6649 h: 10615-13792	l: 1175-2547 h: 3018-7853
	S1mmBY	corrected for B and YS	idem, with force corrected for buoyancy at 1 mm and 'recovered' yield stress	l: 3265-5319 h: 9440-12427	l: 762-2057 h: 2626-7348
	intcept	**Intercept (at H = 1 m)**	Calculation from the linear part of the log stress vs. log height curve	l: 6.2-21.7 h: 22.1-66.3	l: -115--67.2 h: -140--46.9
	intceptBY	corrected for B and YS	idem, with force corrected for buoyancy a.f.o. height and 'recovered' yield stress	l: 0.0004-2.7 h: 3.5-18.6	l: -236--178 h: -190--70.7
	slope	**Slope**	Calculation from the linear part of the log stress vs. log height curve	l: -0.991--0.842 h: -0.927--0.776	l: -0.878--0.712 h: -1.135--0.789
	slopeBY	corrected for B and YS	idem, with force corrected for buoyancy a.f.o. height and 'recovered' yield stress	l: -2.383--1.110 h: -1.174--0.945	l: -1.270--0.972 h: -1.281--0.870
	Ecompr	**Energy of compression (Pam)**	Area of compression peak, representing the energy required for compression	l: 8.6-13.2 h: 20.3-28.4	
	m	**Alternative flow index**	Calculated from the ratio of S1mmBY at both compression speeds (Corradini *et al.* 2000a; see Chapter 5)	0.25-0.40	0.41-0.60
	n0.75	**Constant strain flow index**	Calculated as the slope of viscosity at 0.75 strain for 0.25 and 5 mm/s, assuming lubricated flow (see Chapter 5)	0.21-0.37	0.23-0.38
Relaxation	S1min	**Stress at 1 min (Pa)**	Extracted from stress relaxation data	l: 862-1987 h: 1135-2692	l: 410-714 h: 400-764
	S1minBY	corrected for B and YS	idem, with force corrected for buoyancy at 1 mm and 'recovered' yield stress	l: 43-211 h: 56-284	l: 6-39 h: 11-46
	S2min	**Residual stress (Pa)**	Stress after 2 min at 1 mm, extracted from measurement data	l: 807-1922 h: 1079-2598	l: 396-680 h: 381-725
	Syield	**'Recovered' yield stress (Pa)**	Residual stress at 2 min minus buoyancy stress at 1 mm	l: 602-1718 h: 874-2393	l: 92-381 h: 38-375
Decompression	Smin	**Minimum decompression stress (Pa)**	Peak stress during decompression	l: 4758-6836 h: 14321-18637	
	Edecompr	**Energy of decompression (Pam)**	Area of decompression peak < 0 N, representing the energy required for decompression	l: 9.1-15.7 h: 27.5-38.6	
	Mstick	**Product sticking on probe (g)**	Weight of product stuck to the probe after the measurement	l: 2.8-21.3 h: 18.3-29.8	

*the code for each parameter is in the text preceded by l or h, indicating measurement at low (0.25 mm/s) or high (5 mm/s) compression speed.

Shear measurements

Power-law flow indices and yield stresses in shear were determined from flow-curve measurements. These were performed on a Paar Physica MCR 300 (Anton Paar Benelux, Sint-Martens-Latem, Belgium), equipped with a 40 mm plate/plate system on which sandpaper (kor 80) was attached to prevent slip. After lowering the upper plate and removing the excess of sample, samples were allowed to relax and acclimatize for 5 minutes (custards) or 10 minutes (mayonnaises) at 22°C before the measurements were made. Flow curves were obtained from 0.01-1000 s^{-1}, with 36 measuring points taken logarithmically, each after 15 s. Measurements were made in duplicate or triplicate. Yield stress behavior of mayonnaises was characterized by fitting the flow curves with the Herschel-Bulkley model. For custards, the Casson model was applied, because it gave a better fit. More details on the measurement and calculations are described in Chapter 3.

Sensory assessment

Sensory scores were assessed by a trained sensory panel using a quantitative descriptive (QDA) analysis protocol (Stone & Sidel 1985). The set of mayonnaises and the set of custards were assessed separately. The panel consisted of ten young and healthy subjects who were selected for their above average sensory acuity. Panelists were trained in the use of sensory odor , flavor , texture and afterfeel attributes with samples similar to those later used for measurement. They were paid for their participation. Panel testing took place at the sensory facilities of TNO Quality of Life (Zeist, The Netherlands).

Samples of one product type were assessed in three 2-hour sessions preceded by a training session. All products were presented once per session in a randomized order. During the three sessions, held at the same day and time at 3 consecutive weeks, subjects were presented with three replicates of each sample. Subjects were seated in sensory booths with appropriate ventilation and lighting. During presentation of a sample, subjects first smelled the product and rated odor attributes. Then, subjects took one spoonful of the sample and rated mouthfeel and flavor attributes. After swallowing, the subjects rated afterfeel and aftertaste attributes. Attributes were rated on a computer screen using a mouse and a 100-point visual analog scale anchored at the extremes. Acquisition of the panelist's responses was done by computer using FIZZ software (Biosystemes 1998, v1.20K, France). The attributes and their definitions (see Chapter 3) had been previously generated using a QDA-protocol and are applicable for mayonnaises as well as custards. A more detailed description of the procedures has been given by de Wijk *et al.* (2003b).

Data analysis

Parameters obtained in the squeezing flow measurements and the shear measurements were averaged over the replicates. Sensory scores for each attribute were averaged over all replicates and panel members. Single factor ANOVA analysis ($p < 0.05$) was performed (Microsoft Excel 97 SR-2 software) to test the parameters on their discriminative ability between the products. Relationships within and between the sensory attributes, the squeezing flow parameters and the parameters from the shear measurement were established univariately by Pearson correlations (Microsoft Excel 97 SR-2 software) and in a multivariate way using Principle Component Analysis (PCA) and Partial Least Square Analysis (PLS1) (The Unscrambler 7.6 SR-1, Camo Asa, Oslo, Norway). In univariate analysis, significant correlations were checked and omitted if they were caused by groups or outliers. For multivariate analysis, sensory scores and rheological parameters were mean centered and normalized by their standard deviation.

Results and Discussion

Squeezing flow parameters

Measured ranges of the parameters obtained from the different steps of the squeezing flow measurement are given in Table 6.1 for both product types at both compression speeds. In general, the measurements were very reproducible, with standard deviations between the replicates of the parameters of less than 10%. ANOVA ($p < 0.05$) showed that nearly all parameters could discriminate between the samples within one product type. Stresses measured during the compression step (e.g., at 1 or 2 mm compression) are much higher for mayonnaises than for custards, as well as the residual stress after stress relaxation (see Table 6.1). For this reason, the contribution of buoyancy forces plays a minor role for mayonnaises, while for custards buoyancy forces are in the same order of magnitude as the 'recovered' yield stress. The values found for the stresses during the compression and relaxation step are in agreement to those reported in literature (Suwonsichon & Peleg 1999; Corradini *et al*. 2000a; Corradini & Peleg 2005).

Many of the parameters measured during the compression and decompression step are different for the two (de)compression speeds, similarly to previous observations (see Chapter 5). For example, the stress at 1 or 2 mm compression increases with compression speed (see Table 6.1). In some of these cases, the parameters at low and high speed are significantly correlated (not shown). Parameters measured during the relaxation step at low and high compression speed are strongly correlated as well as similar in value (see Fig. 6.2 for yield stress). The different effects of (de)compression speed on parameters from the different steps of the measurements can be explained by the effect of strain rate; stresses measured during the (de)compression step increase with (de)compression speed because strain rate also increases with (de)compression speed. In the relaxation step, no effect of (de)compression speed is found because no strain rate is applied. Although the samples have experienced differences in strain history caused by different compression speeds, this seems to have only a marginal effect on the parameters measured in the relaxation step. This was found for both types of products.

'Recovered' yield stress
A few of the parameters obtained from the squeezing flow measurement can be considered as properties of the material, which are independent from measurement conditions. Yield stress and flow index are two commonly used rheological material properties. In the imperfect squeezing flow measurement, yield stress can be determined as the residual stress after two minutes of stress relaxation, minus buoyancy forces (Corradini *et al.* 2000a). This is not a true yield stress, however, as the sample has already been deformed, but rather a 'recovered' yield stress, reflecting a degree of solidity (Corradini *et al.* 2000a; Corradini & Peleg 2005). Being a property of the material, the values of the 'recovered' yield stress should be independent from the (de)compression speed. This was indeed shown for all the parameters from the relaxation step (see above), for both types of products, and can be seen for yield stress in Table 6.1 and Fig. 6.2.

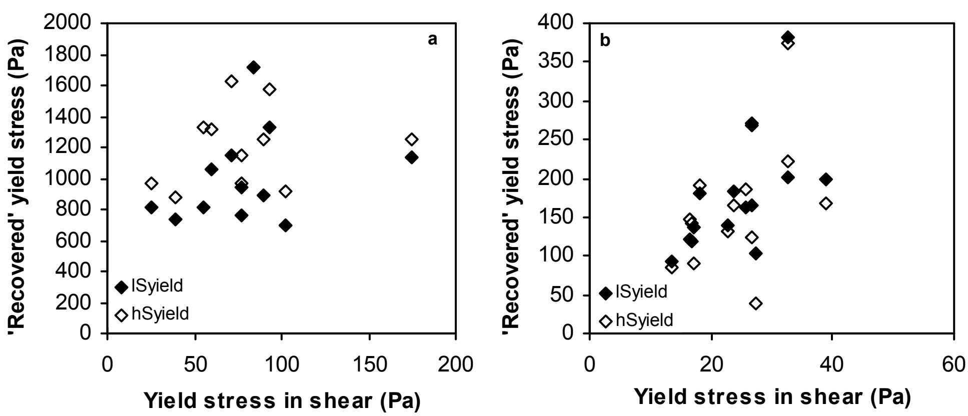

Figure 6.2a,b 'Recovered' yield stress as determined in the squeezing flow measurement versus the yield stress measured in shear, for (a) mayonnaises and (b) custards. Yield stress in shear has been determined from fitting the flow curve with the model of Herschel-Bulkley (mayonnaises) or Casson (custards). Two mayonnaise samples were left out because no yield stress values in shear were available.

Yield stresses obtained from the relaxation step of the imperfect squeezing flow measurements were compared to those from shear measurements, see Fig. 6.2. For both custards and mayonnaises, the values obtained from the squeezing flow measurements are much higher than those obtained from the shear measurements. In addition, yield stresses obtained from the relaxation step of the imperfect squeezing flow measurement and from shear measurements are not clearly related to each other (see Fig. 6.2). Only for custards there is a just significant correlation ($p < 0.05$) with the yield stress measured in the squeezing flow measurement at low compression speed.

Although both types of yield stresses are in agreement to values from the same methods reported in literature (Suwonsichon & Peleg 1999; Štern *et al*. 2001; Tárrega *et al.* 2004; Corradini & Peleg 2005), values obtained from the relaxation step of the squeezing flow measurement are one order of magnitude larger and not clearly related to values obtained from shear measurements. Furthermore, values obtained from the relaxation step of the squeezing flow measurement are also higher than yield stress values obtained from another type of squeezing flow measurement, i.e., a creep test at constant force instead of constant compression speed (Campanella & Peleg 1987c; Meeten 2000). An obvious explanation for the difference in magnitude is that the yield stress obtained from the relaxation step of the squeezing flow measurement was determined after the sample has undergone large-scale deformation. As the product types under study are (pseudo-) thixotropic, this deformation causes the structure to breakdown. Only a part of this structure breakdown is reversible, and only when the sample has rested long enough after the compression step. Because of this, one would expect that the 'recovered' yield stress is lower than the yield stress obtained from the start of deformation. The results in this study show, however, the opposite, so this explanation is invalid. It seems, at least for the product types in this study, that the 'recovered' yield stress obtained from the relaxation step of the imperfect squeezing flow measurement at constant compression speed, introduced by Corradini *et al*. (2000a), reflects a different material property than the conventional yield stress.

Flow indices
The other material property that can be obtained from squeezing flow measurements is the flow index, which can be determined by different methods. In case of ideal squeezing flow, the flow index in elongational flow can be calculated from the slope of the log stress versus log height curve (see Fig. 6.1b). Depending on whether the conditions are fully lubricated or nonlubricated, the flow index is equal to -slope or to -½(slope+1), respectively. In Chapter 5, where the imperfect squeezing flow technique was explored for custards and mayonnaises, it was shown that the slope of the log stress versus log height curve does not give a valid flow index in the current measurement set-up. The slope was shown to depend on compression speed and the calculated flow index, assuming lubricated or nonlubricated conditions showed nonrealistic values. This was attributed to the presence of a certain amount of friction, (pseudo-) thixotropic and yield-stress behavior of the samples and instrumental artifacts due to the imperfect set-up.

The results in the current study on the slopes of the log stress versus log height curve confirm these conclusions: values of the slope are different for low (l-) and high (h-) compression speed and/or cannot be calculated into realistic flow indices (see Table 6.1). When lubricated flow is assumed, flow indices calculated from the slopes range around and above 1 (not shown), which is more typical for the behavior of a Newtonian material than for a strain-thinning material. Assuming nonlubricated conditions leads in a few instances to negative values for the flow index (not shown), which is highly unlikely from a theoretical point of view. These observations are true for both types of products and for the slope as such as well as for the slope corrected for buoyancy and yield stress (slopeBY, see Table 6.1).

Table 6.2. Values of flow index in shear and significant univariate Pearson correlations ($p < 0.01$) with squeezing flow parameters associated with flow index.

		In shear n (power law)	
		Mayonnaise (n = 0.09-0.22)	Custard (n = 0.13-0.29)
In compression	lslope hslope lslopeBY hslopeBY	ns	-0.57--0.72
	m	0.67	ns
	n0.75	0.76	ns

In Chapter 5, two alternatives for the determination of the flow index were used, both combining squeezing flow data measured at different compression speeds. The alternative flow index m (Corradini *et al.* 2000a) is calculated as the ratio of compression stress minus relaxation stress at two different compression speeds (see Table 6.1). The validity of m as flow index is limited because it only compensates for the effect of yield-stress behavior and buoyancy forces (see Chapter 5). The thixotropy-corrected flow index n0.75 compensates for the (pseudo-) thixotropic character of custards and mayonnaises. To accomplish this, the effect of strain history is eliminated by comparing results measured at different compression speeds at the same strain. N0.75 was calculated in this paper as the slope between viscosities at 0.75 strain of the curves at both compression speeds (see Table 6.1). Values of both m and n0.75 are, similar to those found in Chapter 5, in the range expected for strain-thinning materials (see Table 6.1). The values of m for mayonnaises are also comparable to those reported by Corradini *et al*. (2000a). The meaning and validity of both flow index alternatives is, however not very clear. Comparing the different versions of the flow index obtained from the imperfect squeezing flow measurement with the one in shear can shed some light on the issue of the meaning and validity of the different flow indices. Flow indices measured in elongational deformation are known to be different from those in shear, but we do expect values in the same range, as both should reflect strain-thinning behavior. Table 6.1 and 6.2 show that m and the thixotropy-corrected flow index n0.75 are, for mayonnaises, not only similar in value but also significantly related to the flow index in shear, see also Fig. 6.3. For custards (Table 6.1 and 6.2), m and n0.75 are similar in value to flow indices in shear, but not significantly related. Significant correlations exist only with the slopes from the log stress versus log height curve, which were shown above to be invalid as flow indices in elongational flow. These correlations for custards have no physical meaning, but can be explained by their mutual strong correlations with fat content (not shown).

So, the results on the different ways to determine the flow index from squeezing flow measurements show that m and n0.75 could represent the flow index in elongation, as values are in the expected range. But it is hard to state which of both, if any, truly represents the flow index in elongation, because only for mayonnaises they are related to flow indices in shear. Furthermore, both flow index alternatives should be regarded with care, as other factors affecting the squeezing flow measurement, such as a certain amount of friction and measurement artifacts are not compensated in their calculation. Additionally, n0.75 is in this study only based on measurements at two different compression speeds.

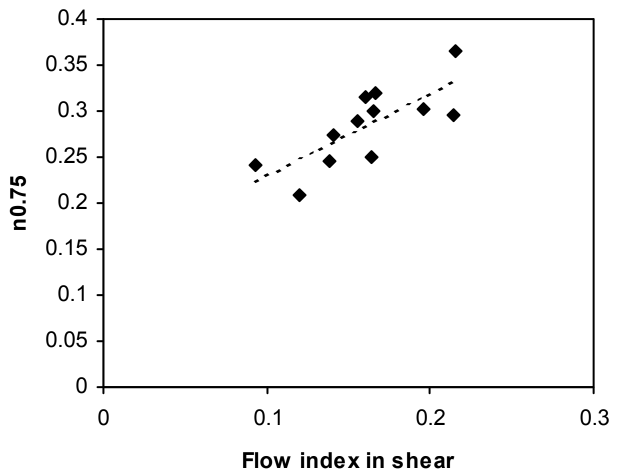

Figure 6.3 Thixotropy-corrected flow index at 0.75 elongational strain from the squeezing flow measurement versus the power law flow index measured in shear for mayonnaises. The Pearson correlation coefficient of the relation is 0.76 (see Table 6.2).

Relations with sensory perception

Relations between sensory attributes and squeezing flow parameters were analyzed by univariate correlations. Table 6.3 and 6.4 present the main results. Because of the large differences in sensory and squeezing flow properties, mayonnaises and custards were analyzed separately. The relations between attributes and parameters, among sensory attributes and among squeezing flow parameters were also visualized by multivariate PCA biplots (Fig. 6.4 and 6.6), as well as the location of the individual samples. It should be noted that, for both types of products, the variance explained by PC1 and PC2 is not very large (see Fig. 6.4 and 6.6). This is because the relations among and between sensory attributes and squeezing flow parameters are rather complicated and difficult to represent in a plot with two axes. Adding a third axis (PC3, 13% for mayonnaises and 15% for custards, not shown) does improve the representability only to a limited extent. Because the general outline of the PCA's are in agreement with the univariate results, the PCA's are yet shown to give a general insight in the relations among and between sensory attributes and squeezing flow parameters.

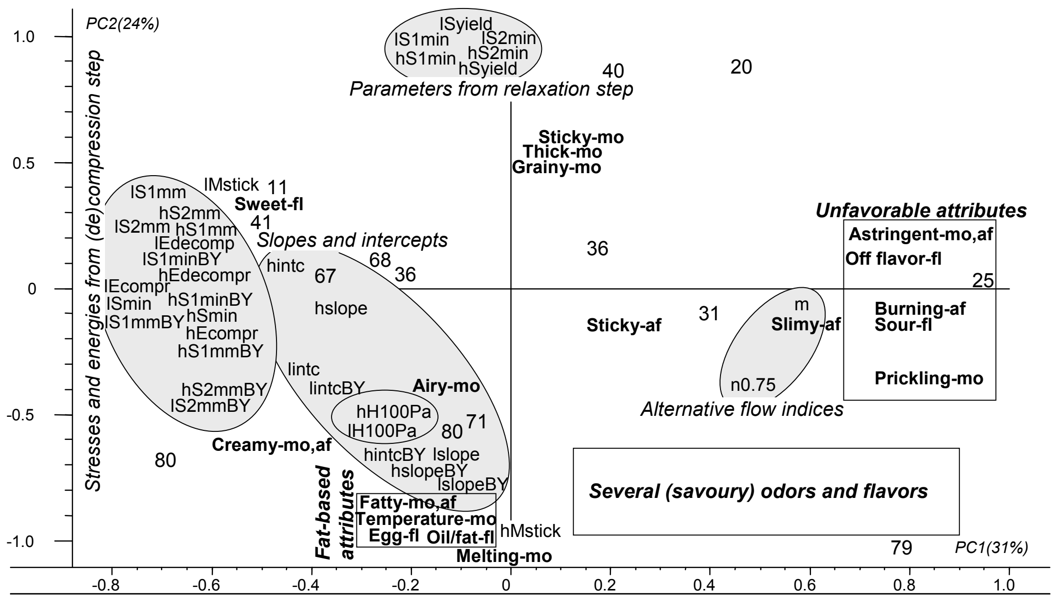

Figure 6.4 PCA biplot of sensory attributes and parameters from the squeezing flow measurement for mayonnaises. The relative location of the samples is indicated by their fat content. PC3, not shown, explains 13% of variance and is roughly made up by fat-based attributes versus grainy mouthfeel. The affix -mo, -fl, or -af of the sensory attributes stands for mouthfeel, flavor or afterfeel, respectively. For the sake of clarity, some attributes with limited importance have been omitted and mouthfeel and afterfeel of the same attribute have been combined when they are strongly correlated. For explanation of the squeezing flow parameters, see Table 6.1.

Mayonnaises

Results show that the mayonnaise samples can be described by two sensory dimensions. The first dimension consists of nontextural attributes such as sweet flavor and a group of unfavorable attributes (e.g., astringency, prickling and off flavor). These attributes play a prominent role in the data set, as they make up the first Principal Component in the PCA of Fig. 6.4. The second dimension includes mouthfeel and afterfeel attributes describing texture and form a diagonal axis from left under to upper right in the PCA. Among these attributes, creaminess, a group of fat-based attributes (e.g., fattiness and temperature mouthfeel) and melting mouthfeel are significantly related to each other, and the latter is negatively correlated to thickness and other viscosity-related attributes (see Fig. 6.4). The relative location of individual mayonnaise samples in the PCA can be attributed to their scores on unfavorable attributes in combination with their scores on fat-based attributes. Samples were perceived as creamy when they had high scores on fattiness (i.e., had high fat contents) and low scores on the unfavorable attributes. Also the squeezing flow parameters can be classified in groups based on univariate correlations, similar to the groups shown in the PCA of Fig. 6.4. This classification is a reflection of the type of parameter and the step in the squeezing flow measurement from where it was obtained. Parameters describing stresses and energies during the compression and decompression step at low compression speed make up one large group, and similar parameters at high compression speed another group. In Fig. 6.4 they are combined into one group. Both parameter groups are especially correlated to the unfavorable attributes, in a negative manner, see Table 6.3 and Fig. 6.4. Other groups, such as the one comprising the 'recovered' yield stress and other parameters from the relaxation step and the one consisting of both flow indices (i.e., m and n0.75) have limited significant correlations with sensory (texture) attributes (see Table 6.3), although Fig. 6.4 suggests otherwise. The same is true for a group of parameters describing slopes and intercepts of the log stress versus log height curve, with and without correction for buoyancy and yield stress (see Table 6.3).

Table 6.3 Main significant univariate Pearson correlations ($p < 0.05$) of sensory attributes with squeezing flow parameters for mayonnaises, per attribute. The affix -mo, -af, -fl, -od stands for mouthfeel, afterfeel, flavor or odor, respectively.

	max | r |	Correlating parameter (group)
Creamy-mo,af	0.84	lH100Pa (and hH100Pa)
	-0.59	hSmin (and others from group stresses & energies during (de)compression step at high compression speed)
	0.56	hMstick
Fat-based attributes (temperature-mo, fatty-mo, fat-af, oil/fat-fl, egg-fl)	0.92	hMstick
	0.77	hH100Pa (and lH100Pa)
Melting-mo	0.72	lH100Pa
	0.67	hMstick
Airy-mo	ns	
Thick, sticky-mo	ns	
Grainy-mo	0.77	lH100Pa (and hH100Pa)
Unfavorable attributes (prickling-mo, burning-af, astringent-mo,af, off-fl, sour-fl)	-0.76	hSmin (and others from group stresses & energies during (de)compression step at high compression speed)
	-0.71	lSmin, lS1mmBY (and others from group stresses & energies during (de)compression step at low compression speed)
Sweet-fl	0.58	lSmin
Slimy-af	0.62	lSmin (and lS2mm, lEcompr)
Sticky-af	ns	
Other flavors and odors (intensity-fl,od, herbs-fl,od, salt-fl,at, off-od, sour-od)	-0.73	lSyield (and others from group relaxation step)
	0.72	hMstick

Creaminess is strongly correlated to parameters representing the sample height at 100 Pa, especially to the one measured at low compression speed (lH100Pa), see Table 6.3 and Fig. 6.4 and 6.5. This parameter is obtained from the beginning of the compression step. The relation with lH100Pa is also the main relationship found by multivariate PLS1 prediction for creamy mouthfeel (slope = 0.68, r = 0.78). This result was not significantly improved when several squeezing flow parameters were combined in one prediction; only a slightly better prediction was obtained for the combination of lH100Pa with a parameter describing stress or energy during (de)compression, e.g., lS2mmBY (slope = 0.68, r = 0.82). Other combinations of parameters gave much weaker predictions for creamy mouthfeel. Fat-based attributes are very strongly correlated to hMstick (see Table 6.3 and Fig. 6.4), the amount of product sticking onto the probe after the experiment at high compression speed. This parameter is also strongly correlated to fat content (r = 0.91). Correlations with melting mouthfeel (Table 6.3) are moderately in quality and overlap with correlations found for creaminess and fat-based attributes. This suggests that the relations found for melting mouthfeel are caused indirectly via the correlation of melting with creaminess and fat-based attributes. For thickness and other viscosity-related attributes no significant univariate correlations were found (see Table 6.3), as thickness varied to a limited extent in the mayonnaise sample set.

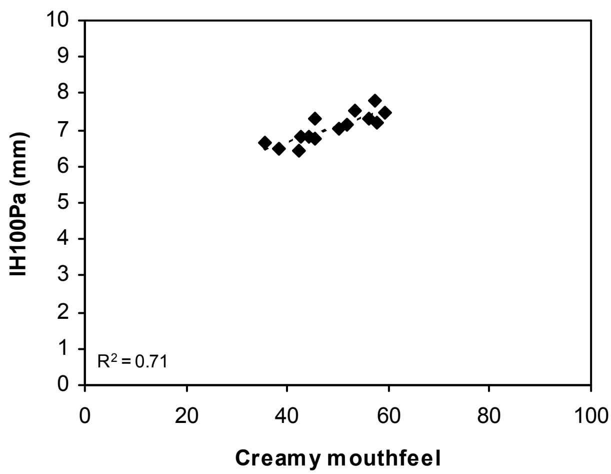

Figure 6.5 Relation of creaminess with lH100Pa for mayonnaises. LH100Pa is the height of the sample under the probe when the compression force exceeds 100 Pa.

The results show that the main mouthfeel and afterfeel attributes of mayonnaises correlate moderately to very strongly to squeezing flow parameters, but that the two most relevant parameters do not represent rheological behavior during compression. HMstick was obtained from the decompression step but is not correlated to other parameters from this step. The parameter is mainly governed by adhesive properties, which are strongly affected by the fat content of the samples. High fat samples seem to adhere more to the hydrophobic Teflon probe than low-fat samples. LH100Pa, describing the height of the sample when the stress exceeds 100 Pa during compression at low speed, is determined at the beginning of the compression step. This parameter is moderately related to fat content and seems to reflect the penetration resistance of the product and/or its density. The penetration distance is inversely related to lH100Pa; in a tougher product, the compression force will exceed 100 Pa at smaller penetration depths, thus at larger lH100Pa. Other parameters reflecting resistance of a sample to deformation, such as the slope determined from the compression step or the yield stress in shear, are, however not significantly correlated to lH100Pa or creaminess. Differences in product density can also lead to differences in lH100Pa, because the container was filled with a constant mass of sample. When a product has a relative large density, the initial sample height will be small, so the compression force will exceed the low threshold of 100 Pa after longer traveling of the probe, giving a smaller value for lH100Pa. Exact product densities were not determined in this study, but the range found for lH100Pa (Table 6.1) corresponds to a range in densities of 813-1013 kg/m^3. As this is a realistic range for a set of mayonnaises including full-fat as well as low-fat mayonnaises (Elert 2005), density differences between products are able to explain the variation in lH100Pa. So, creaminess of mayonnaises in this study was best predicted by a parameter that seems to reflect mainly density. This correlation is much stronger than correlations with fat content ($r = 0.75$) or squeezing flow parameters reflecting rheological properties. Mayonnaise samples in this study were perceived as creamy when their density was low.

Custards

Relations between sensory attributes and squeezing flow parameters for custards are given in Table 6.4 and Fig. 6.6. Creamy and fatty mouthfeel and afterfeel make up the first dimension, being univariately correlated. Both are not significantly univariately correlated to melting mouthfeel, although the PCA suggests otherwise. A second dimension is made up by melting and airy mouthfeel, which are negatively univariately correlated to thickness and other viscosity-related attributes, as well as to a group of unfavorable attributes (see Fig. 6.6). Individual custard samples are mainly distributed according to their fat content; custards with the highest fat contents are located on the creaminess-side of PC1. Both samples with 1.8% fat and based on soy-milk instead of milk score relatively high on the unfavorable attributes, locating them close to these attributes in the right upper quadrant. The parameters from the squeezing flow measurement can be classified in similar groups as those described for mayonnaises, but correlation between the groups is stronger than in case of mayonnaises. This is also visible in Fig. 6.6; although the sensory attributes are widely distributed over the PCA, the groups of squeezing flow parameters are located closely to each other on the first principal component. Only a limited number of significant univariate correlations were found between attributes and squeezing flow parameters (see Table 6.4). As the only significant univariate correlations of squeezing flow parameters are negative correlations with creaminess, fattiness and related flavors, the parameter groups cluster together opposite these attributes (see Fig. 6.6).

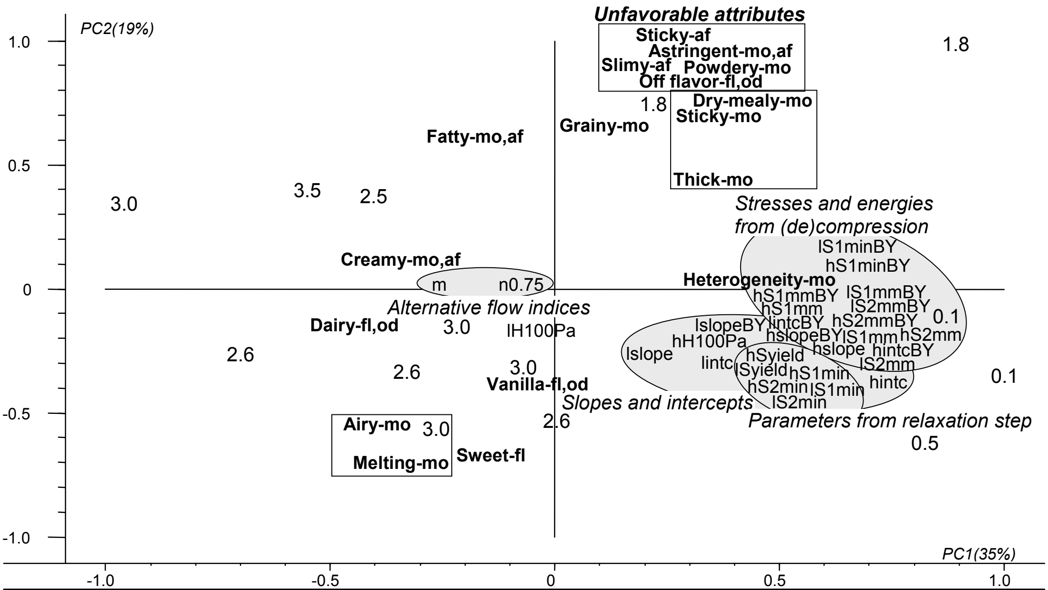

Figure 6.6 PCA biplot of sensory attributes and parameters from the squeezing flow measurement for custards. The relative location of the samples is indicated by their fat content. PC3, not shown, explains 15% of variance. The affix -mo, -fl, or -af of the sensory attributes stands for mouthfeel, flavor or afterfeel, respectively. For the sake of clarity, some attributes with limited importance have been omitted and mouthfeel and afterfeel of the same attribute have been combined when they are strongly correlated. For explanation of the squeezing flow parameters, see Table 6.1.

Creaminess and fattiness are not significantly correlated to the whole cluster of parameter groups in Fig. 6.6, but mainly to the group of slopes and intercepts at low and high compression speed (see Table 6.4 and Fig. 6.7). Univariate correlations and multivariate PLS1 prediction yielded three types of parameters important for the prediction of creamy mouthfeel for custards: the slope of the log stress versus log height curve at high compression speed, preferably corrected for yield stress and buoyancy (i.e., hslopeBY), the same parameter measured at low compression speed (i.e., lslopeBY) and a parameter describing the buoyancy and yield stress-corrected stress after 1 min of relaxation (i.e., lS1minBY). These three parameters are moderately univariately correlated to creaminess (see Table 6.4) and in combination could predict creaminess well by PLS1 (slope = 0.67, r = 0.80). When one of the parameters is left out, a reasonable prediction still remains. Together, they describe the behavior of the custard samples during compression and subsequent relaxation. Only two out of three, hslopeBY and lS1minBY are strongly correlated to fat content of the samples (r = 0.84). Correlations of fatty mouthfeel and fat afterfeel with squeezing flow parameters partly overlap with those found for creaminess, but are somewhat weaker (see Table 6.4). The other attributes, including melting, thickness and other viscosity-related attributes show no significant correlations with squeezing flow parameters (see Table 6.4) and were poorly predictable by a combination of parameters in PLS1.

Table 6.4 Main significant univariate Pearson correlations ($p < 0.05$) of sensory attributes with squeezing flow parameters for custards, per attribute. The affix -mo, -af, -fl, -od stands for mouthfeel, afterfeel, flavor or odor, respectively.

	max r	
Creamy-mo,af	-0.77 -0.74 -0.73	hslopeBY (and others from group slopes and intercepts at high compression speed) lslopeBY, lintcBY (and others from group slopes and intercepts at low compression speed) lS1minBY (and hS1minBY)
Fatty-mo, fat-af	-0.71 -0.63	hslopeBY, hslope (and others from group slopes and intercepts at high compression speed) lintc (and others from group slopes and intercepts at low compression speed)
Melting, airy	ns	
Sticky-mo *Dry/mealy*	ns	
Thick-mo	ns	
Grainy-mo	ns	
Heterogeneity-mo	ns	
Unfavorable attributes (astringent-mo,af, off-od,fl, powdery-mo, sticky-af)	ns	
Sweet-fl	ns	
Slimy-af	ns	
Dairy-od,fl	-0.86	lS1minBY (and others from group stresses during compression step at both compression speeds)

So, the study of custards shows that squeezing flow parameters are poorly related to sensory attributes, except for creaminess, fattiness and related flavors. Creaminess of custards was found to be negatively related to the (negative) slopes of the stress increase during compression at low and high compression speed, as well as to the buoyancy and yield stress-corrected stress obtained 1 min after the compression has stopped. Both slope parameters reflect the increase in sample resistance at increasing strain rate. The other parameter is equal to the stress decay between 1 and 2 min of relaxation, i.e., the part of the stress at 1 min that can be dissipated by the sample, in contrast to the 'recovered' yield stress and buoyancy, which are still present after 2 min of relaxation. This parameter is not significantly related to other parameters of the relaxation step, but rather to parameters describing stresses during the compression step. A custard sample was perceived as creamier when it showed a steep increase in stress during compression (at low and high speed), as well as limited stress decay after halting the compression. Fat content of the custard samples can only partly explain this behavior. From the two squeezing flow parameters that were found to be relevant for mayonnaises, hMstick was not determined for custards and lH100Pa showed no significant correlations. This latter result can be explained by the fact that differences in initial sample heights and densities were small in the custards study (not shown). The limited number of custard attributes that correlate to squeezing flow parameters could be due to the absence of the effect of saliva in the squeezing flow measurement. Janssen *et al*. (2007) showed that parameters describing enzymatic breakdown are important for viscosity-related attributes such as melting and thickness of custards.

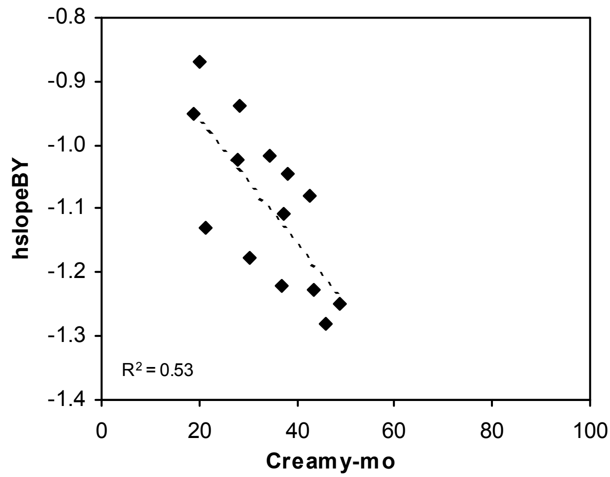

Figure 6.7 Relation of creaminess with hslopeBY for custards. HslopeBY is the slope of the log stress versus log height curve at high compression speed.

Implications for applicability, oral processing and perception

Mayonnaises

By comparing relations of texture attributes with squeezing flow parameters and those found previously with parameters from shear measurements, the applicability of imperfect squeezing flow measurements in relation to texture perception can be established, as well as the relevance of elongational flow in oral processing and perception. For mayonnaises, comparison on the same sample set (see Chapter 3) shows that univariate correlations with squeezing flow parameters are equally strong (in r) as those found with parameters from small and large shear deformation measurements. In Chapter 3, creaminess and other attributes were mainly related to the stage of shear deformation where the structure breaks up and the sample starts to flow. This is different from the results of creaminess with the squeezing flow measurement, where the most prominent parameter (lH100Pa) might be regarded as describing the sample in a relatively undisturbed state. But it is more likely that lH100Pa and the other important parameter, hMstick have no direct rheological background, but are indirectly correlated to texture attributes via correlations with product density and fat content. Because there are to be no strong, direct correlations between important mouthfeel and afterfeel attributes and parameters obtained during the (de)compression and relaxation step for the mayonnaises in the current study, imperfect squeezing flow measurements seem not a relevant addition (or replacement) to measurements of mayonnaises in shear. On similar grounds, one may draw conclusions about the conditions relevant for oral perception. As the squeezing flow measurement in the current set-up operates under a combination of elongational and shear deformation, it seems that elongational deformation and elongational properties play a marginal role compared to shear during oral perception of mayonnaises. Although it is clear that compression movements do take place during oral processing, the effects of elongation appear to be of limited relevance for the perception of sensory attributes. This was also concluded in Chapter 2, where a model including the compression movement did not improve predictions for perceived thickness compared to a model that only described the shear movement. It can not be precluded however, that the marginal effect of elongational deformation on sensory perception as found in this study might have been caused by the choice of mayonnaise samples and/or the selected set-up of the measuring technique.

Custards

For custards, parameters obtained during the compression and relaxation step of the imperfect squeezing flow measurement are related to creaminess and fattiness, but not to other mouthfeel and afterfeel attributes. The quality of the prediction of creaminess by squeezing flow parameters is comparable to that by parameters from small and large shear deformation measurements (Janssen *et al.* 2007). So, squeezing flow measurements, combining elongational and shear deformation, may be a useful addition to small and large deformation measurements in shear for these attributes. Combined analysis of shear and squeezing flow data should disclose whether the measurements are complementary or overlap. Correlations of creaminess and fattiness of custards with shear and squeezing flow parameters are only moderately in quality because both measurements lack saliva-induced breakdown of starch, which is important for the perception of custards. Results on different compression speeds show that parameters measured at high compression speeds (5 mm/s) have more and relatively stronger relations with texture attributes. This could be because the high compression speed is more similar to oral conditions than the low one. Another explanation is that the contribution of shear is increased with compression speed and that the contribution of shear in the compression movement is more relevant than the contribution of elongation. In any case, it is clear that shear measurements cannot be replaced by squeezing flow measurements, because the latter are poorly related to attributes such as thickness, stickiness and melting. Regarding oral conditions during perception of sensory attributes, one may conclude that elongational behavior during oral compression movements plays a role in oral processing and sensory perception of custards, next to shear, especially for the perception of creaminess and fattiness. The squeezing flow parameters found to relate with creaminess suggest that during oral compression of custards the change in stress (i.e., the slope) is more relevant for perception than the absolute value of the stress.

As shown throughout this paper, mayonnaises and custards behave differently regarding squeezing flow parameters, sensory attributes, their relations and implications thereof. This can be explained by the fact that both types of products, although both semisolids, differ largely from each other. One example is the difference in flavor and odor profile, which is known to affect the perception of creaminess (de Wijk *et al.* 2006a). But other texture attributes were perceived differently as well. An important difference between both types of products is the relevance of saliva-induced breakdown in the oral processing and perception; because the starch-based matrix of custards is more strongly affected by enzymatic breakdown than the matrix of mayonnaises, breakdown by saliva is much more important for the perception of custards than it is for mayonnaises. Additionally, both sample sets used in this study are very different in character; the set of custards is dominated by differences in thickness, while the mayonnaise samples are characterized by large differences in fat content at comparable thicknesses.

Conclusion

The imperfect squeezing flow technique is an easy and practical method that is sensitive enough to distinguish differences within custard and mayonnaise samples. The measurement technique is able to determine properties that are related to sensory attributes. The quality of these relations is, in general, comparable to that found for shear measurements. For custards, squeezing flow properties are mainly related to creaminess and fattiness attributes. Other attributes are poorly predicted due to the lack of saliva-induced breakdown in the measurement. Custards were perceived as creamier when the product showed a steep increase in stress during compression as well as limited stress decay afterwards. As creaminess and fattiness are related to parameters from the compression step of the squeezing flow measurement, one may conclude that elongational deformation plays a role next to shear during oral perception of these attributes for custards. Imperfect squeezing flow measurements of custards, especially at high compression speed can therefore be a useful addition to measurements in shear. For mayonnaises, the main texture attributes are strongly related to the height of the sample in the beginning of compression, when the compression force exceeds 100 Pa and to the amount of sample sticking onto the probe after the measurement. These parameters have a background in density and fat content rather than in rheological properties. Perceived thickness and other viscosity-related attributes are not significantly related to any parameter from the squeezing flow measurement. Both observations for mayonnaises can be partly explained by the fact that differences in thickness for the mayonnaises under study were much smaller than differences in fat content. So, for the currently applied measurement technique and the current sample set, it seems that elongational deformation and elongational properties are less relevant for oral processing and perception of mayonnaises and imperfect squeezing flow measurements are less useful than shear measurements. Investigations on the meaning and validity of the material properties measured with the imperfect squeezing flow technique showed that the 'recovered' yield stress, obtained from the relaxation step, is one order of magnitude larger and not clearly related to values obtained from other methods in shear or elongation. This suggests that, at least for the product types in this study, the 'recovered' yield stress, as introduced by Corradini *et al*. (2000a) reflects a different material property than the conventional yield stress. From the three different ways to obtain a flow index from the squeezing flow measurement, the values obtained directly from the slope of the log stress versus log height curve of the compression step are no true material properties. Both alternative versions, one corrected for (pseudo-) thixotropic behavior and one corrected mainly for yield stress and buoyancy may, however represent a valid flow index in elongation.

Acknowledgements

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7

MAIN FINDINGS FOR MAYONNAISES AND CUSTARDS

In this chapter, the results of the previous chapters are combined and discussed. The conclusions are presented as a list of main findings covering multiple chapters. These findings were primarily obtained for mayonnaise samples, as custards were only used in Chapter 2, 5 and 6.

Mayonnaises

Shear rheological measurements are very useful instrumental techniques to measure oral texture perception of mayonnaises

Chapter 2, 3 and 4 showed that shear rheological measurements at small, oscillating- and large, continuous deformation are very useful instrumental techniques to measure oral texture perception of mayonnaises. Properties obtained in these measurements could predict the perception of most of the texture attributes well, by modeling (Chapter 2), by univariate correlations (Chapter 3), and by multivariate predictions (Chapter 3 and 4). Mouthfeel attributes, perceived during oral processing were better predicted by rheological measurements than afterfeel attributes, perceived after swallowing.

The rheological measurements that proved to be most useful are the dynamic stress sweep measurement, primarily the nonlinear region of this measurement, and both measurements in large deformation, i.e., the steady shear-rate measurement and the flow-curve measurement. These measurements were performed from 0.1-1000 Pa at 1 Hz, at 10-1 s^{-1}, and from 0.01-1000 s^{-1}, respectively. The perception of thickness could be modeled well by a physical-physiological model that only required properties measured in the flow-curve measurement, next to some parameters describing oral physiology. Measurements at small, oscillating deformations in the linear region (LVER) showed a limited number of relations with texture attributes. So, the linear region of the dynamic stress sweep measurement and the dynamic frequency measurement, which was measured completely in the linear region are of limited use. It is remarkable that the complex mechanisms of oral processing and texture perception can be represented relatively well by one single type of properties, reflecting behavior in deformation.

The nonlinear region of the dynamic stress sweep measurement, especially the behavior at 500% strain, is most successful in measuring properties related to texture perception

Parameters obtained from the nonlinear region of the dynamic stress sweep are the most important rheological parameters in relation to texture attributes of mayonnaises (Chapter 3 and 4). These parameters describe the behavior of the mayonnaises during transition from a (viscoelastic) solid 'at rest' to a (viscoelastic) fluid that flows and subsequent behavior in 'dynamic', oscillating flow. The disruption of the sample structure that is required for this transition is distinctively different for samples with high and low fat content. The structure of mayonnaises with high fat contents, determined primarily by a fat-droplet network, breaks down suddenly and rapidly. The structure of mayonnaises with low fat contents, determined primarily by a hydrocolloid thickener matrix, breaks down more gradually over a broader (oscillating) stress range. This difference in behavior is especially expressed in the parameter measured in dynamic flow, i.e., tan δ at 500% (oscillating) strain. This parameter, though obtained from a region of the measurement where the dynamic deformation is not perfectly sinusoidal, is the most successful rheological parameter in relation to oral texture perception and is strongly related to fat content and composition of the thickener phase. The combination of tan δ at 500% and tan δ at 50 or 250 Pa fully captures the behavior at the start of flow. These latter parameters reflect properties during transition to flow and are also relevant in relation to oral texture perception.

Next to parameters from the nonlinear region of the dynamic stress sweep, also specific parameters from rheological measurements in large shear deformation were found to be relevant in relation to texture attributes. One of the primary parameters obtained from these measurements is the viscosity at high shear stress of 500 s^{-1}, measured by a flow curve. In addition, concaveness C describes the curvature of the flow curve and is related to the type and amount of carbohydrate thickener. The viscosity decay constant k, obtained from the steady shear-rate measurement reflects the amount of pseudo-thixotropic breakdown of the sample and is strongly related to fat content.

Other measurement techniques, such as the squeezing flow measurement and novel instrumental measurements only slightly improve the relations with texture attributes
For a complete prediction of texture attributes, additional properties, other than those obtained by shear rheometry, are required, but these were not obtained by one of the other measurement techniques applied in the studies. Replacement of shear rheological measurements by the squeezing flow measurement or addition of novel instrumental measurements improved the relations with oral texture perception of mayonnaises only slightly.

With the imperfect squeezing flow technique in a Teflon geometry, rheological properties of semisolids under conditions of (partial) elongational flow can be measured in an easy and practical way (Chapter 5 and 6). However, the measurement does not determine pure elongational behavior, the material properties obtained are different from those obtained by shear measurements and results should be regarded more qualitative than quantitative. Furthermore, Chapter 5 showed only weak correlations between texture attributes of commercial mayonnaises and squeezing flow parameters that had a rheological background. Only indirect measured properties, reflecting density and fat-related adhesive properties showed strong relations with texture attributes. Therefore, the measurement of rheological properties under partly elongational deformations by the squeezing flow set-up used in Chapter 5 and 6 seems to be of limited use for the replacement of rheological measurements in shear.

In another study (Chapter 4), several novel, qualitative physicochemical measurements that determine nonrheological physicochemical properties and/or reflect changes of properties of the food during oral processing were combined with shear rheological measurements to investigate whether the predictions of texture attributes improved. Only measurements reflecting the effect of saliva and friction measurements were to some extent successful when added to shear rheological measurements. For textural afterfeel attributes the improvement of the prediction was large, but for textural mouthfeel attributes the improvement was only small. Measurements on the turbidity of rinse water after swallowing and viscosity measurements including saliva were most successful of the novel instrumental measurements. Turbidity measurements reflect the composition of the sample and the effect of saliva on it. Viscosity measurements including saliva or water, performed with a specially developed structure breakdown cell, reflect the dilution effect of saliva as well as the effect of enzymatic breakdown on starch. Parameters reflecting the general effect of saliva or water on viscosity are the most relevant. Friction measurements in addition to shear rheological measurements are only useful to some extent for a few attributes related to the perception of particles, such as graininess. Due to the set-up of the study, it could only be established that the addition of these novel measurements to shear rheological measurements is to a some extent useful, but not whether these novel measurements are able to replace rheological measurements. Measurements on the fat droplet size were shown to be not relevant in relation to texture attributes (Chapter 4).

Creaminess is related to melting, fattiness and the absence of unfavorable sensations
Sensory studies in this thesis showed that the studied mayonnaises are perceived as creamy when they score high on melting and fat-based attributes. On the other hand, heterogeneity mouthfeel and trigeminal and flavor unfavorable sensations, such as prickling mouthfeel, astringency mouth- and afterfeel, off flavor and sour flavor have a negative effect on the perception of creaminess. Mouthfeel and afterfeel of creaminess are strongly related. Although many publications, for a wide range of semisolid food products, show that creaminess is related to perceived thickness, this relationship was not found for the studied mayonnaises. Instead, melting mouthfeel was found to be important for the perception of creaminess. This attribute was often negatively related to thickness mouthfeel, but it is not the opposite, because it is more complex and surface-related. The deviation between the results in this thesis and literature can be partly explained by the fact that the relationship of creaminess with thickness mouthfeel and with melting mouthfeel probably shows an optimum rather than a linear relation, and that thickness was only varied in one of the studies. Creaminess relates positively with the amount of fat in mayonnaises, hence also with fat-based attributes such as fattiness and temperature mouthfeel. Specific thickeners,

i.e., xanthan, cause the mayonnaise to be perceived as heterogeneous, which reduces the perception of creaminess.

Creaminess is strongly related to rheological behavior at the start of flow
Creaminess of mayonnaises is strongly related to their rheological behavior at the start of flow, when the initial structure is disrupted and forced to flow (see above). For this reason, tan δ at 500% strain is the most important parameter for the perception of creaminess. Both the attribute and the parameter are strongly affected by the composition of the samples. Next to rheological properties in dynamic flow, also properties describing the transition to flow and properties in large deformation flow are relevant. A mayonnaise is perceived as creamy when the structure breakdown at the start of flow happens very sudden and rapid, but afterwards, during large deformation flow, viscosity does not decrease further, with a limited amount of (pseudo-) thixotropic breakdown. Addition of parameters reflecting the effect of saliva, i.e., from turbidity measurements on rinse water or from viscosity measurements including saliva improved the predictability of creamy mouthfeel to a small extent. Creamy afterfeel could be predicted from similar rheological and other physicochemical parameters as creamy mouthfeel, but the quality of the prediction from shear rheological measurements alone was moderately when samples differed in more than fat content. Addition of parameters reflecting the effect of saliva was required to predict creamy afterfeel well.

Properties in the predictions of fatty mouthfeel, fat afterfeel and other fat-based attributes for mayonnaises are strongly related to fat content. Relations of these attributes with properties from rheological and novel instrumental measurements are similar in quality and with more or less the same parameters as relations of creamy mouthfeel and afterfeel.

Thick mouthfeel of mayonnaises is a bulk attribute that could be predicted well by a relative simple physical-physiological model that predicts the shear stress perceived on the tongue at simplified oral processing conditions (Chapter 2). The only material properties required in this model are the flow index and consistency, describing the rheological behavior in large shear deformation. Next to these and other properties measured in large deformation flow, also properties describing the transition to flow and in dynamic flow contributed to the multivariate prediction of thickness and other viscosity-related attributes (Chapter 4). Thick samples stay firm up to high dynamic stresses and viscosities in large deformation flow remain high as well. Properties reflecting rheological behavior including elongation or the effect of saliva or other physicochemical properties do not play a role in the prediction of thickness for mayonnaises (Chapter 4 and 6). This can be explained by the fact that thickness is perceived as one of the first mouthfeel attributes, probably before saliva has started to take effect.

Melting mouthfeel of mayonnaises is, in its relations to physicochemical properties, more similar to creaminess and fattiness than to thickness (Chapter 3, 4 and 6). This attribute could be predicted well from rheological parameters in shear similar to those mentioned for creaminess. One particular shear rheological property in relation to melting is the viscosity at high deformation rates, at 500 s^{-1}. Samples that are perceived as melting start to flow at relative low dynamic stresses, but in large deformation flow viscosity decreases to a limited extent. Parameters reflecting the effect of saliva on viscosity also play a role for melting.

These results on the relations between texture attributes and physicochemical properties and among texture attributes can be considered as general applicable for mayonnaises. The relationships among the texture attributes and with rheological properties were first established by modeling and by univariate correlation on a small group of commercial mayonnaise samples with large variation in fat content and limited variation in perceived thickness (Chapter 2 and 3). These relationships were later confirmed by multivariate analysis in another study on a larger and broader sample set of model- and commercial mayonnaises (Chapter 4). Although many of the most relevant rheological parameters were in the univariate study related to the amount of fat, they were still found to be relevant when the sample set contained a majority of model mayonnaises with equal fat contents and differences in thickener phase.

The relations between texture attributes and physicochemical properties are in general assumed to be linear, but in some cases they appeared to be more complex (Chapter 2 and 3). The relationship between thickness and the modeled shear stress on the tongue was found to be semilogarithmic. Also, when sensory attributes were correlated to shear rheological parameters, in some instances the logarithmic of the parameter performed better than the as such or the power-law version. Quadratic relationships, in which attributes show an optimum with underlying physicochemical properties were not tested, but will certainly be present in some cases.

Perception of all mouthfeel attributes requires a certain amount of oral deformation, but elongational deformation is less relevant than shear deformation

As the relationships between texture attributes and physicochemical properties are presumably causal, these relationships can be translated to oral processing conditions during perception of textural mouthfeel and afterfeel attributes (Chapter 2,3,4 and 6). As shear rheological properties are the most relevant properties in relation to all the textural mouthfeel attributes of mayonnaises, it seems that rheological behavior due to the deformation of the food bolus in the mouth is sensed by the oral tissue and translated to perception of these attributes. All assessed mouthfeel attributes of mayonnaises, including those perceived immediately after food intake such as thickness, require a certain minimal amount of shear deformation of the food bolus in the mouth, because initial rheological properties ('at rest') did not relate to mouthfeel attributes (Chapter 3 and 4). For similar reasons, the results of Chapter 2 and 6 seem to suggest that the compression movement and elongational deformation play a minor role to shear deformation in perception, although they are included in oral processing. The results in Chapter 3 and 4 suggest that the level of oral shear deformation required for the perception of mouthfeel attributes consist of deformation conditions at which the structure is disrupted and the mayonnaises start to flow as well as conditions of large deformation at rates up to 500 s^{-1}. The rheological behavior of the food bolus at the start of flow is sensed and translated into perceived fat content and creamy mouthfeel. Friction properties and the effect of saliva on properties of the food bolus and oral coating are also sensed, but they play a smaller role in the resulting perception of textural mouthfeel attributes. Their role is more relevant for textural afterfeel attributes, because oral deformation is minimal during perception of these attributes.

Custards

Studies on custards in this thesis are limited to modeling of thickness from a physical-physiological model (Chapter 2) and relating elongational properties from squeezing flow measurements to texture attributes (Chapter 5 and 6). Both studies used commercially available custards only. More elaborate studies on commercial- and model custards have been performed and published in the project which this thesis is part of (de Wijk *et al.* 2003b; Jellema *et al.* 2005; de Wijk *et al.* 2006b; Janssen *et al.* 2007), with the author of this thesis involved as coauthor. The results in these other studies confirm the observations of Chapter 2, 5 and 6 and investigate these further. For this reason, the other, published studies are also included in the next sections.

There are distinct differences in perception within the group of semisolid foods; custards behave differently from mayonnaises

Comparing the results of custards and of mayonnaises shows that there are distinct differences in perception within the group of semisolid foods, as custards behave differently from mayonnaises. These differences can be attributed to the differences in composition of both types of products; custards contain much less fat, which is also varied less than for mayonnaises. In custards, the carbohydrate thickener phase is the main structural component. This phase consists primarily of starch. The dispersed phase of fat droplets plays a limited role. Another relevant difference is the dairy character of custards.

These differences in composition and structure result in very different sensory perceptions and different relations of creaminess with other sensory attributes (Chapter 6, de Wijk *et al.* 2003b; Jellema *et al.* 2005; de Wijk *et al.* 2006b). Creaminess is less related to fat content and fat-based attributes, but more to characteristics of the thickener-phase and related attributes such as roughness, heterogeneity and thickness. Creaminess of custards was negatively related to roughness and heterogeneity. It was positively related to thickness, instead of melting in case of mayonnaises. This difference is probably due to the optimum in the relation of creaminess with thickness and melting; custards are on the other side of the optimum than mayonnaises. Another important dimension in the sensory make-up of custards is the thick versus melting axis. Nontextural attributes that affect creaminess are also different from those of mayonnaises; pleasant flavors and odors such as dairy and vanilla contribute to a creamier custard (de Wijk *et al.* 2006b), while trigeminal and flavor attributes such as prickling, astringency and sour-flavor contribute to a less creamy mayonnaise (Chapter 3).

The saliva-induced breakdown of starch, absent in rheological measurements is more essential for the perception of custards than it is for mayonnaises

The differences in composition and structure between custards and mayonnaises also result in different relations between texture attributes and properties from physiological measurements. Only some of the mouthfeel attributes of custards could be predicted satisfactorily from shear rheological measurements alone (Jellema *et al.* 2005; Janssen *et al.* 2007). The physical-physiological model (Chapter 2) predicted thickness less well for custards than for mayonnaises. Rheological properties under conditions of (partial) elongational flow from the squeezing flow measurement (Chapter 6) were stronger related to texture attributes of custards than to texture attributes of mayonnaises, but these relations are still not satisfactorily. These results can for a large part be explained by the lack of the effect of saliva in the properties obtained in the rheological measurements. Custards are much more sensitive to saliva-induced breakdown of starch because its structure relies more on starch. Mayonnaises, even those with low fat contents are less sensitive, probably because they contain also non-starch thickeners. Structure breakdown cell measurements (Janssen *et al.* 2007) are very relevant for the prediction of texture attributes of custards. Also friction measurements play a important role for custards, as the creaminess versus roughness axis reflects primarily lubrication properties. The importance of friction measurements can be explained by the low fat contents of custards compared to mayonnaises. Because of this, lubrication is not yet at its maximum in case of custards and increases with fat content (de Wijk & Prinz 2005). A last difference between custards and mayonnaises concerns the type of shear rheological properties important for the predictions of texture attributes of custards; next to properties describing the transition to flow, in dynamic flow and at large deformation, also rheological properties 'at rest' from the linear regime of the dynamic stress sweep measurement are relevant for perception (Jellema *et al.* 2005; Janssen *et al.* 2007).

SUMMARY

SAMENVATTING

Summary

To produce healthy foods that we like to eat, it is essential to understand how sensory perception of food relates to food properties. Much of the research on the origins of sensory perception has been done for taste, smell and odor properties. The work in this thesis focuses on oral texture perception of semisolid foods, in particular the sensory property creaminess. This work was part of a large multidisciplinary project of the Wageningen Centre of Food Sciences, in which sensory science, oral physiology and material science were combined. The aim of the work in this thesis was to identify physicochemical properties underlying oral texture perception of semisolid foods, in particular creaminess, as well as to find instrumental measurements other than bulk shear rheometry that can measure these properties. With these results, the origins of oral texture attributes and the relevance of specific instrumental measurements could be established, as well as oral mastication conditions during perception of semisolids. To achieve this aim, mayonnaises and custards were subjected to assessments by sensory panels and to various instrumental measurements.

Mayonnaises and custards are semisolid food products. These type of products show complex, viscoelastic behavior, displaying solid-like as well as liquid-like properties. During eating, food products are subjected to oral processing conditions, including oral movements and the effect of saliva. The initial food properties, changes to these properties caused by oral processing and properties of the food retained in the mouth after swallowing are sensed by sensors in the mouth and nose and translated in the brain into perception of a range of sensory attributes. Among them are texture attributes, which are associated with structural, mechanical and surface properties of the food bolus and oral coating. Creaminess is a complex but important texture attribute, as it is highly appreciated by consumers. The sensory perception of a food product can be assessed by quantitative descriptive analysis. Physicochemical properties of the food products in this thesis were primarily measured by bulk shear rheometry, which determines properties associated with deformation and flow of materials. Other measurements applied in this thesis determine other types of physicochemical properties such as friction and/or properties under mouth-like conditions, especially the effect of saliva.

In **Chapter 2**, models that describe the mechanical deformation in the mouth were investigated in order to predict orally perceived thickness of mayonnaises and custards. To this end, the applicability of the physical-physiological models of Kokini *et al*. (1977), calculating the shear stress on the tongue, were tested and attempts were made for improvements. The results showed a relationship between shear stress on the tongue and thickness, in accordance with the work of Kokini *et al*. (1977), but this relationship was only linear within a limited range of shear stresses (mayonnaises < 150 Pa; custards < 30

Pa). Beyond this range, the linear relationship breaks down and the thickness levels off with shear stress for both mayonnaises and custards. The relationship over the entire range of shear stresses found in this study could be satisfactorily described by a semilogarithmic (Fechner's) relation. Comparison of both models of Kokini *et al*. (1977) showed that the quality of the thickness prediction by the decreasing-height model, including lateral and compression movements of the tongue, and the constant-height model, simulating lateral movements only, was similar, for both types of products. So, the relative simple constant-height model was sufficient to predict perceived thickness. For most mayonnaises, the contribution of the lateral movement of the tongue to the shear stress in the decreasing-height model was orders of magnitude larger than the contribution of the squeezing or compression movement of the tongue towards the palate. This difference in magnitude is affected by the low value measured for the compression force and by the high values for material consistency K, due to neglect of yield-stress behavior. For custards, both models of Kokini *et al.* (1977) were found to be less adequate. It was proposed that this is because the models ignore interactions with saliva. Several routes to improve the modeling of both types of products by incorporating viscoelastic behavior were unsuccessful.

Chapter 3 and 4 identified relationships between texture attributes and physicochemical properties for mayonnaises. **Chapter 3** explored the relations of texture attributes with parameters describing bulk rheological behavior in shear. Rheological characterization consisted of different types of small and large deformation measurements, followed by extensive parameterization of the curves. Relations were established for a set of commercial mayonnaises (11-80% fat, limited variation in thickness) and in a univariate and multivariate way. The large variation in fat content played a prominent role in the results. Sensory analysis showed that creaminess was positively related to temperature mouthfeel and melting mouthfeel and negatively to unfavorable sensations. Parameters from dynamic stress sweep measurements (i.e., tan δ at 500% strain), describing structure breakdown at the start of flow, correlated best to sensory attributes, in particular to creaminess and fat-based attributes. Mayonnaises were perceived as creamier when they showed a sudden and rapid structure breakdown at the start of flow. For melting and stickiness, viscosity at high shear rates was also important. As many of the parameters and attributes were strongly affected by fat content, their relationships could be indirect instead of causal.

In **Chapter 4**, the general applicability of the results of Chapter 3 was tested for a larger and broader set of model and commercial mayonnaises and by multivariate prediction of all texture attributes. The sample set varied in fat content as well as the type and amount of thickener. In addition, mayonnaises were characterized by novel instrumental measurements that cover other physicochemical properties and/or reflect changes of the food properties during oral processing. Predictions of texture attributes by rheometry combined with these novel instrumental measurements were compared to predictions by rheological parameters alone to establish whether these extra measurements are a useful addition to shear rheometry. Results showed that most of the texture attributes were well predicted by rheological parameters alone. For many attributes, such as creaminess, fattiness and melting, parameters from the novel instrumental measurements played a complimentary though small role. Predictions of most of the afterfeel attributes were strongly improved by the addition of parameters from other instrumental measurements. The most important measurements in relation to texture attributes were rheological measurements at large deformation and measurements in the nonlinear regime of dynamic stress sweep and two novel instrumental measurements reflecting the effect of saliva: turbidity measurements of rinse water and viscosity measurements in the structure breakdown cell with added saliva. Friction measurements were only useful for attributes directly related to particles. Relations between texture attributes and bulk rheological parameters confirmed the results of Chapter 3 and can therefore be regarded as generally applicable for mayonnaises: tan δ at 500% strain, reflecting the extent of fluid-like character of the samples during high strain dynamic flow, is the parameter that describes best the property underlying creaminess and other texture attributes. Properties from other types of rheological measurements are also relevant to the perception of texture attributes, except for the initial rheological properties obtained in the LVER.

Chapter 5 and 6 investigated the relevance of bulk rheological properties in elongation, obtained by the imperfect squeezing flow technique (Corradini *et al.* 2000), in relation to the oral texture perception of mayonnaises and custards. In **Chapter 5** the technique of imperfect lubricated squeezing flow in a Teflon geometry was explored for the measurement of elongational behavior of mayonnaises and custards. Two Newtonian products, one of low (0.07 Pas) and one of high (18 Pas) shear viscosity were used as references. Measurements of custards and mayonnaises did not behave according to either the theory of lubricated or nonlubricated squeezing flow, as there were effects of the initial sample height and compression speed. Also, calculated values for the flow index were not as we had expected. The same was true for the Newtonian samples. An important factor explaining the effect of compression speed was the presence of a certain amount of friction, rendering both lubricated theory as well as nonlubricated theory nonapplicable. Correcting for (pseudo-) thixotropic behavior of custards and mayonnaises appeared to be an effective way of obtaining realistic values for the flow index. The presence of buoyancy also affected the results, especially in case of low viscous products and the effect of initial sample height. Other factors that played a role in the results were yield stress of custards and mayonnaises and instrumental artifacts associated with the imperfect set-up of the measurement, especially for the highly viscous products. Quantitatively correcting the results for all of these factors was not possible at this point. Although imperfect squeezing flow measurements in a Teflon geometry is a very practical way to measure semisolids such as custards and mayonnaises under (partly) elongational deformation, results should be regarded more qualitative than quantitative.

In **Chapter 6**, the technique of imperfect squeezing flow measurements in a Teflon geometry was applied to characterize a set of commercial mayonnaises and a set of commercial custards. Values of parameters extracted and calculated from the compression, relaxation and decompression step were compared to properties measured in shear and were related to sensory attributes in both a univariate and multivariate way. Results showed that the squeezing flow method is sensitive enough to distinguish differences within the set of custard and mayonnaise samples. For custards, squeezing flow properties were mainly related to creaminess and fattiness. Other attributes were poorly predicted due to the lack of saliva-induced breakdown in the measurement. Custards were perceived as creamier when the product showed a steep increase in stress during compression and limited stress decay after halting the compression. For mayonnaises, attributes were strongly related to parameters reflecting density and fat content rather than rheological properties. This was partly due to the limited differences in thickness of the mayonnaises under study. For custards, imperfect squeezing flow measurements seem useful next to shear measurements, especially for the attribute creaminess, and elongational deformation seems relevant for oral processing and perception. For the mayonnaises in this study, this is less true, given the currently applied measurement technique. Investigations on the meaning and validity of the material properties measured in the imperfect squeezing flow measurement showed that the 'recovered' yield stress from the squeezing flow measurement reflects a different material property than the conventional yield stress. From the three different ways to obtain a flow index from the squeezing flow measurement, only the constant-strain approach and the alternative flow index approach may lead to a valid flow index in elongation.

Chapter 7 combined and discussed the results of the previous chapters. The conclusions are presented as a list of main findings, primarily obtained for mayonnaises. Shear rheological measurements are very useful instrumental techniques to measure oral texture perception of mayonnaises. The nonlinear region of the dynamic stress sweep measurement, especially the behavior at 500% strain, is most successful in measuring properties related to oral texture perception. Other measurement techniques, such as the squeezing flow measurement and novel instrumental measurements only improved relations with texture attributes slightly. Creaminess of mayonnaises is related to the attributes melting, fattiness and the absence of unfavorable sensations. Its perception is strongly related to the rheological behavior of the mayonnaises at the start of flow. Thick mouthfeel of mayonnaises can be predicted well by a relative simple physical-physiological model that predicts the shear stress perceived on the tongue at simplified oral processing conditions. Perception of all mouthfeel attributes requires a certain minimal amount of oral deformation, but elongational deformation is less relevant than shear deformation. Comparing sensory perception and relations with physicochemical properties of custards with that of mayonnaises shows that there are distinct differences within the group of semisolid foods. The saliva-induced breakdown of starch, absent in conventional rheological measurements is more essential for the perception of custards than it is for mayonnaises.

In conclusion, this thesis has shown that, especially for mayonnaises, texture perception can be predicted relatively well by bulk rheological properties in shear and that the application of other types of measurements investigated in this thesis in addition to bulk shear rheometry is of limited use.

Samenvatting

Om gezonde én lekkere levensmiddelen te maken, is het van essentieel belang om het verband te begrijpen tussen de sensorische waarneming en de eigenschappen van die levensmiddelen. Veel onderzoek op dit gebied is gedaan voor de waarneming van smaak en geur. Het werk in dit proefschrift onderzoekt de waarneming van textuur in de mond bij dikvloeibare levensmiddelen, met name die van de sensorische eigenschap romigheid. Dit onderzoek was onderdeel van een groot, multidisciplinair project van het Wageningen Centre of Food Sciences, waarin sensorische wetenschappen, orale fysiologie en materiaalkunde werden gecombineerd. Het doel van het onderzoek in dit proefschrift is om de fysisch-chemische eigenschappen aan te wijzen die ten grondslag liggen aan orale textuur waarneming, in het bijzonder romigheid, en om instrumentele technieken naast bulkreologische metingen onder afschuiving te vinden die deze eigenschappen kunnen meten. Met deze resultaten wordt de oorsprong van orale textuur attributen en de relevantie van specifieke instrumentele metingen bepaald, maar ook de verwerkingsomstandigheden in de mond tijdens het eten en waarnemen van dikvloeibare levensmiddelen. Om dit doel te bereiken, werden mayonaises en vla's onderworpen aan beoordelingen door een sensorisch panel en aan instrumentele metingen.

Mayonaises en vla's zijn dikvloeibare levensmiddelen. Dit soort produkten vertonen complex, viscoelastisch gedrag, met zowel vaste-stofachtige als vloeistofachtige eigenschappen. Tijdens het eten wordt een voedselprodukt onderworpen aan orale manipulatie, zoals bewegingen in de mond en het effect van speeksel. De initiële produkt eigenschappen, veranderingen van deze eigenschappen als gevolg van orale manipulatie en de eigenschappen van het produkt dat achterblijft in de mond na doorslikken worden gevoeld door de sensoren in de mond en neus en worden in de hersenen vertaald in waarneming van verschillende sensorische attributen. Dit zijn onder andere textuur attributen, attributen die te maken hebben met mechanische, structuur- en oppervlakte-eigenschappen van de voedselbolus en de mondcoating. Romigheid is een gecompliceerd maar belangrijk attribuut, omdat het door consumenten sterk gewaardeerd wordt. De sensorische waarneming van een voedselprodukt kan worden gekarakteriseerd door quantitatieve beschrijvende analyse. Fysisch-chemische eigenschappen van voedselprodukten werden in dit proefschrift voornamelijk gemeten met bulkreologische metingen onder afschuiving, waarmee eigenschappen worden gemeten die te maken hebben met vervorming en het stromen van materialen. Andere metingen in dit proefschrift bepalen andere soorten fysisch-chemische eigenschappen zoals frictie en/of eigenschappen onder mondachtige omstandigheden, met name het effect van speeksel.

In **Hoofdstuk 2** werden modellen die mechanische vervorming in de mond beschrijven onderzocht op hun voorspellende waarde voor de orale waarneming van dikte van

mayonaises en vla's. Hiertoe werd de toepasbaarheid van de fysisch-fysiologische modellen van Kokini *et al*. (1977), die de afschuifspanning op de tong berekenen, getest en werd gepoogd deze te verbeteren. De resultaten lieten zien dat er een relatie is tussen de afschuifspanning op de tong en de waarneming van dikte, in overeenstemming met het werk van Kokini *et al*. (1977), maar dat deze relatie alleen lineair is onder een bepaalde waarde van de afschuifspanning (voor mayonaises < 150 Pa; voor vla's < 30 Pa). Boven deze afschuifspanning is de relatie niet meer lineair maar vlakt de diktewaarneming af bij toenemende afschuifspanning, voor zowel mayonaises als vla's. De relatie over de gehele bandbreedte aan afschuifspanning zoals gevonden in deze studie kon goed worden beschreven met een semilogaritmische (Fechner) relatie. Het vergelijken van beide modellen van Kokini *et al*. (1977) liet zien dat de kwaliteit van de voorspelling van dikte door het 'afnemende hoogte'-model, met daarin laterale en compressiebewegingen van de tong, en het 'constante hoogte'-model, waarin alleen laterale bewegingen gesimuleerd worden, gelijkwaardig zijn, voor beide typen produkten. Oftewel, het relatief simpele 'constante hoogte'-model was voldoende om de waargenomen dikte te voorspellen. Voor de meeste mayonaises was de bijdrage van de laterale beweging van de tong aan de afschuifspanning in het 'afnemende hoogte'-model vele malen groter dan de bijdrage van de pers- of compressiebeweging van de tong richting verhemelte. Dit verschil in grootte wordt beïnvloed door de lage waarde die is gemeten voor de compressiekracht en door de hoge waardes voor de materiaal consistentie, K als gevolg van het verwaarlozen van de aanwezigheid van zwichtspanning. Voor vla's werd gevonden dat beide modellen van Kokini *et al.* (1977) minder geschikt zijn. Er wordt gesteld dat dit komt doordat de modellen de interactie met speeksel negeren. Verschillende manieren om het modeleren van beide typen produkten te verbeteren door het meenemen van viscoelastisch gedrag hadden geen succes.

Hoofdstuk 3 en 4 zoeken naar relaties tussen textuur attributen en fysisch-chemische eigenschappen van mayonaises. **Hoofdstuk 3** onderzocht de relaties van textuur attributen met parameters die bulkreologisch gedrag onder afschuiving beschrijven. De reologische karakterisering bestond uit verschillende types metingen, bij kleine en grote vervormingen, gevolgd door uitgebreide parameterisatie van de curves. De relaties werden bepaald voor een groep commercieel verkrijgbare mayonaises (11-80% vet, beperkte variatie in waargenomen dikte), op zowel univariate als multivariate wijze. De grote variatie in vetgehalte speelde een prominente rol in de resultaten. Sensorische analyse liet zien dat romigheid positief was gerelateerd aan temperatuur-mondgevoel en smeltend-mondgevoel en negatief aan onprettige sensaties. Parameters van de 'dynamic stress sweep' meting (met name tan δ bij 500% vervorming) die de instorting van de structuur bij het begin van stroming beschrijven correleerden het beste met de sensorische attributen, met name romigheid en de attributen gebaseerd op vet. Mayonaises werden als romiger waargenomen als zij een plotselinge en snelle instorting van hun structuur lieten zien aan het begin van stroming. Voor smeltend en plakkerig mondgevoel was ook de viscositeit bij hoge afschuifvervorming belangrijk. Omdat veel van de parameters en attributen sterk worden beïnvloed door vetgehalte, zouden hun relaties indirect in plaats van causaal kunnen zijn.

In **Hoofdstuk 4** werd de algemene toepasbaarheid van de resultaten van Hoofdstuk 3 getest met een grotere en bredere groep van model- en commercieel verkrijgbare mayonaises en door middel van multivariate voorspelling, voor alle textuur attributen. De groep monsters varieerde in vetgehalte, maar ook in type en hoeveelheid verdikker. Daarnaast werden de mayonaises ook gekarakteriseerd met nieuwe instrumentele metingen die andere fysisch-chemische eigenschappen omvatten en/of veranderingen van de voedseleigenschappen tijdens orale manipulatie weergeven. Voorspellingen van textuur attributen door een combinatie van reologische en deze nieuwe metingen werden vergeleken met voorspellingen door alleen reologische parameters om vast te stellen of deze extra metingen een bruikbare toevoeging zijn op reologische metingen onder afschuiving. De resultaten lieten zien dat de meeste textuur attributen goed werden voorspeld met alleen reologische parameters. Voor veel attributen, zoals romigheid, vettigheid en smeltend-mondgevoel, speelden parameters van de nieuwe instrumentele metingen een aanvullende, maar kleine rol. Voorspellingen van de meeste nagevoel attributen werden wel sterk verbeterd door toevoeging van parameters van andere instrumentele metingen. De meest belangrijke metingen in relatie tot textuur attributen waren reologische metingen bij grote vervormingen, metingen in het nonlineaire gebied van de 'dynamic stress sweep' meting en twee nieuwe instrumentele metingen die het effect van speeksel weergeven: turbiditeitsmetingen van mondspoelwater en viscositeitsmetingen in de structuur-afbraak-meetcel met toegevoegd speeksel. Frictiemetingen waren alleen nuttig voor attributen die direct gerelateerd zijn aan deeltjes. Relaties tussen textuur attributen en bulkreologische parameters bevestigden de resultaten van Hoofdstuk 3 en kunnen daarom als algemeen toepasbaar worden beschouwd voor mayonaises: tan δ bij 500% vervorming, die de mate van vloeibaarachtig karakter van de monsters weergeeft tijdens dynamische stroming bij hoge vervorming, is de parameter die het best de eigenschap beschrijft die ten grondslag ligt aan romigheid en andere textuur attributen. Eigenschappen van andere soorten reologische metingen zijn ook relevant voor de waarneming van textuur attributen, behalve dan de initiële reologische eigenschappen verkregen in het LVER.

Hoofdstuk 5 en 6 onderzochten de relevantie van bulkreologische eigenschappen in rek, verkregen met de 'imperfect squeezing flow' techniek (Corradini *et al.* 2000), in relatie tot de orale textuur waarneming van mayonaises en vla's. In **Hoofdstuk 5** wordt de 'imperfect squeezing flow' techniek in een Teflon geometrie onderzocht voor het meten van rekgedrag van mayonaises en vla's. Twee Newtonse produkten, één met lage (0.07 Pas) en één met hoge (18 Pas) viscositeit onder afschuiving werden gebruikt ter referentie. Metingen aan vla's en mayonaises gedroegen zich niet volgens de theorie van de gelubriceerde of de niet-gelubriceerde 'squeezing flow', want er bleek een effect van de initiële monster hoogte en van de compressiesnelheid te zijn. Ook waren de berekende waarden voor de flow index niet zoals verwacht. Hetzelfde gold voor de Newtonse monsters. Een belangrijke factor ter verklaring van het effect van de compressiesnelheid was de aanwezigheid van een bepaalde mate van frictie, waardoor zowel de gelubriceerde theorie, als ook de niet-gelubriceerde theorie niet meer van toepassing is. Corrigeren voor het (pseudo) thixotropische gedrag van vla's en mayonaises bleek een effectieve manier te zijn om realistische waarden voor de flow index te krijgen. De aanwezigheid van opwaartse krachten beïnvloedde ook de resultaten, met name in het geval van laag visceuze produkten en in het geval van het effect van de initiële monster hoogte. Andere factoren die een rol speelden in de resultaten waren zwichtspanning voor vla's en mayonaises en instrumentele artifacten die te maken hebben met het imperfecte karakter van de meettechniek, met name voor de hoog visceuze produkten. Getalsmatig corrigeren van de resultaten voor al deze factoren is niet mogelijk op dit moment. Hoewel 'imperfect squeezing flow' metingen in een Teflon geometrie een erg prakische manier is om dikvloeibare produkten, zoals vla's en mayonaises, te meten onder (gedeeltelijke) rekvervorming, moeten de resultaten meer kwalitatief dan kwantitatief beschouwd worden.

In **Hoofdstuk 6** werd de meettechniek toegepast om een groep commercieel verkrijgbare mayonaises en een groep commercieel verkrijgbare vla's te karakteriseren. Waardes van parameters die verkregen en berekend waren uit de compressie-, relaxatie- en decompressiestap van de meting werden vergeleken met eigenschappen die gemeten waren onder afschuiving en werden gerelateerd met sensorische attributen op een univariate en multivariate wijze. De resultaten lieten zien dat de 'squeezing flow' techniek gevoelig genoeg is om verschillen binnen de groep vla's en de groep mayonaise te onderscheiden. Voor vla's waren de eigenschappen uit de 'squeezing flow' meting vooral gerelateerd aan romigheid en vettigheid. Andere attributen werden slecht voorspeld als gevolg van het gebrek aan door speeksel veroorzaakte structuurafbraak in de meting. Vla's werden als romiger ervaren als het produkt een snelle toename in rekspanning liet zien tijdens de compressiestap en een beperkte afname in rekspanning na het stoppen van de compressie. Voor mayonaises waren de attributen sterk gerelateerd aan parameters die eerder dichtheid en vetgehalte weergeven dan reologische eigenschappen. Dit werd gedeeltelijk veroorzaakt door de beperkte dikteverschillen in de groep bestudeerde mayonaises. Voor vla's lijken 'imperfect squeezing flow' metingen bruikbaar naast metingen onder afschuiving, vooral voor het attribuut romigheid, en lijkt rekvervorming relevant voor orale manipulatie en waarneming. Voor de mayonaises in deze studie is dit minder het geval voor wat betreft de huidig toegepaste meettechniek. Onderzoek naar de betekenis en geldigheid van de materiaaleigenschappen die in de 'imperfect squeezing flow' meting bepaald worden liet zien dat de 'zwichtspanning na herstel' uit de 'squeezing flow' meting een andere materiaaleigenschap weergeeft dan de conventionele zwichtspanning. Van de drie verschillende manieren om de flow index te verkrijgen uit de 'squeezing flow' meting, zouden alleen de constante-vervorming benadering en de alternatieve flow index benadering tot een geldige flow index onder rek kunnen leiden.

In **Hoofdstuk 7** werden de resultaten uit de voorgaande hoofdstukken gecombineerd en bediscussieerd. De conclusies hiervan worden gepresenteerd als een lijst met belangrijkste uitkomsten, voornamelijk verkregen voor mayonaises. Reologische metingen onder afschuiving zijn erg nuttige instrumentele technieken om orale textuur waarneming van mayonaises te meten. Het niet-lineaire gebied van de 'dynamic stress sweep' meting, met name het gedrag bij 500% vervorming is het meest succesvol in het meten van eigenschappen die gerelateerd zijn aan orale textuur waarneming. Andere meettechnieken, zoals de 'squeezing flow' meting en de nieuwe instrumentele metingen, verbeterden relaties met textuur attributen slechts een beetje. Romigheid van mayonaise is gerelateerd aan de attributen smeltend-mondgevoel, vettigheid en de afwezigheid van onplezierige sensaties. De waarneming van romigheid is sterk gerelateerd aan het reologische gedrag van de mayonaise bij het begin van stroming. Dik-mondgevoel van mayonaise kan goed worden voorspeld door een relatief eenvoudig fysisch-fysiologisch model dat de afschuifspanning op de tong onder versimpelde condities van orale manipulatie voorspeld. De waarneming van alle onderzochte mondgevoel attributen vereist een bepaalde minimale hoeveelheid orale vervorming, maar rekvervorming is minder relevant dan vervorming door afschuiving. Het vergelijken van de sensorische waarneming en relaties met fysisch-chemische eigenschappen van vla's met die van mayonaises laat zien dat er duidelijke verschillen zijn binnen de groep van dikvloeibare levensmiddelen. De afbraak van zetmeel door speeksel, afwezig in conventionele reologische metingen is essentiëler voor de waarneming van vla's dan het is voor mayonaise.

Ter conclusie, dit proefschrift heeft laten zien dat, vooral voor mayonaises, waarneming van de textuur relatief goed kan worden voorspeld door bulkreologische eigenschappen onder afschuiving en dat de toepassing van andere types metingen die in dit proefschrift zijn onderzocht slecht beperkt nuttig zijn als toevoeging op bulkreologische metingen onder afschuiving.

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List of Publications

Refereed journals

R.A. de Wijk, L.J. van Gemert, **M.E.J. Terpstra** & C.L. Wilkinson, **2003**. Texture of semi-solids; sensory and instrumental measurements on vanilla custard desserts. *Food Quality and Preference* 14, 303.

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Curriculum Vitae

Marjolein Elisabeth Johanna werd op 18 juni 1971 geboren in Hoogezand-Sappemeer en groeide op in Norg. Haar VWO-diploma behaalde zij in 1989 bij het Dr. Nassau College te Assen. In datzelfde jaar begon zij met de studie Scheikunde, later Technische Scheikunde, aan de Rijksuniversiteit Groningen. De focus van haar vakken en afstudeeronderzoek lag daarbij op fysische eigenschappen en extrusie van (bio)polymeren. Na het behalen van haar bul in 1995 verlegde ze haar werkveld richting food en agrotechnologie met een kennisinstandhoudingsplaats bij het toenmalige Instituut voor Agrotechnologisch onderzoek (ATO-DLO, nu Agrotechnology & Food Innovations, A&F) te Wageningen. In 1998 werd zij daar aangenomen als wetenschappelijk onderzoeker in de afdeling voedselextrusie. Tussen 2000 en 2008, beginnend met een korte fase als pre-AIO, deed zij haar promotieonderzoek voor het toenmalige Wageningen Centre for Food Sciences (WCFS, nu Top Institute Food and Nutrition, TIFN), in dienst van de leerstoelgroep Fysica en fysische chemie van levensmiddelen bij de Wageningen Universiteit en met A&F als werklocatie. Een groot deel van de resultaten van dit promotieonderzoek wordt beschreven in dit proefschrift.

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