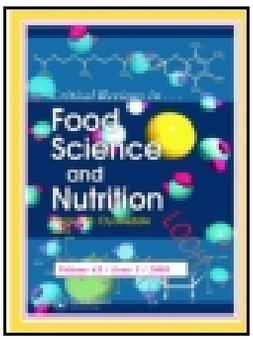

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Squeezing Flow Viscometry for Nonelastic Semiliquid Foods — Theory and Applications

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ASTRACT: In most conventional rheometers, notably the coaxial cylinders and capillary viscometers, the food specimen is pressed into a narrow gap and its structure is altered by uncontrolled shear. Also, most semiliquid foods exhibit slip, and consequently the measurements do not always reflect their true rheological properties. A feasible solution to these two problems is squeezing flow viscometry where the specimen, practically intact and with or without suspended particles, is squeezed between parallel plates. The outward flow pattern mainly depends on the friction between the fluid and plates or its absence ("lubricated squeezing flow"). Among the possible test geometries, the one of constant area and changing volume is the most practical for foods. The test can be performed at a constant displacement rate using common Universal Testing Machines or under constant loads (creep array). The tests output is in the form of a force-height, force-time, or height-time relationship, from which several rheological parameters can be derived. With the current state of the art, the method can only be applied at small displacement rates. Despite the method's crudeness, its results are remarkably reproducible and sensitive to textural differences among semiliquid food products. The flow patterns observed in foods do not always follow the predictions of rheological models originally developed for polymer melts because of the foods' unique microstructures. The implications of these discrepancies and the role that artifacts may play are evaluated in light of theoretical and practical considerations. The use of squeezing flow viscometry to quantify rheological changes that occur during a product's handling and to determine whether they are perceived sensorily is suggested.

KEY WORDS: rheometry, texture, elongational viscosity, biaxial flow.

I. INTRODUCTION

The viscosity or consistency of many foods is a prime textural characteristic and hence an important measure of their quality. It can also affect the efficacy of unit operations in the food industry, notably pumping, filling, and evaporation. Because of the role it plays in heat transfer, it can also affect the safety of many thermally processed foods, such as canned soups, refried beans, and other Mexican dishes. Viscosity measurements therefore are routinely performed in the food industry and research laboratories, primarily as part of quality control or product development, but also in order to create a database for engineering design.

Liquid and semiliquid foods have a wide range of consistencies: from "watery" (e.g., beverages) to almost fully solid (e.g., butter, ice-cream), and consequently their texture is assessed by a variety of methods. The instruments used to determine foods viscosity, or consistency, can be classified in several ways. For our purpose they can be divided into 'empirical' and 'scientific'. The first group includes devices such as the Bostwick and the 'back extrusion cell' (Bourne, 2002). They are 'empirical' because

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what they measure is strongly influenced by their arbitrary geometry. Therefore, it is not clearly defined in physical terms and cannot be expressed in generally accepted units. In contrast, the 'scientific instruments' are particularly designed so that their results can be expressed in terms such as shear stress, shear rate, shear strain, modulus, etc., that is, as universal quantities that are independent, at least in principle, of the sensor's specific design. The most commonly used rheometers of this type are the capillary and coaxial viscometers. In the first type the pressure drop along the capillary is transformed into a shear stress at the wall and the volumetric flow rate to shear rate. In rotational viscometers, having a Couette, parallel plates or cone and plate sensor, the torque is converted into shear stress and the speed of rotation into shear rate, taking into account the sensor's geometry and dimensions. These rheometers can also be used in a dynamic mode, in which a sinusoidally varying strain or stress is applied to determine the fluid's viscoelastic properties. In all the above-mentioned methods the tested material's properties are determined through theoretical rheological models, which combine the fluids constitutive equation and the particulars of the sensor's geometry and test conditions. Less popular in food research, let alone industrial quality control, are rheometers that sense shear and normal forces simultaneously. These are particularly suitable for 'elastic' liquids such as wheat doughs and melted cheeses.

The theoretical relationships between the rheological properties of fluids and their mechanical behavior when subjected to different tests are well understood, and there is a large body of literature that deals with the subject (e.g., Schowalter, 1978; Bird et al., 1987). Food applications of such theories can be found in many review articles and books (e.g., Moskowitz, 1988; Steffe, 1996; Rao, 1999). Most of the basic rheological models that are currently used in food research were originally developed for synthetic polymers melt, non-food emulsions, and suspensions such as paints. Their application to foods having a fragile gel structure such as yogurt or tomato juice concentrates has two serious problems:

1. Almost without exception the tested specimen has to be pressed into the narrow space of the sensor, for example, the narrow gap of a coaxial viscometer. Thus, merely mounting a specimen may disrupt or destroy its internal structure and hence modify its rheological properties. Because the amount of damage is unknown, there is always an element of uncertainty as to whether the measurements truly reflect the intact food's properties and to what extent. The problem is further aggravated when the damage is irreversible, as in yogurt for example, and hence cannot be reversed even by letting the specimen rest for a long time. The obvious solution to this problem is to produce the specimen in the sensor rather than to transfer it from another container. This, however, may be an impractical option in most cases, because of technical and cost considerations.
2. Many foods exhibit what is known as wall slip. Because particles in a shear field created by the flow of a fluid in a tube tend to migrate from the wall region to the tube center, a suspension, almost always, is less concentrated near the wall. Hence, what is in contact with the sensor is not the original material but a layer of liquid with little or no suspended particles. This is equivalent to the presence of a thin film of a lubricant. The result is a plug flow instead of the expected fully developed shear flow on which the shear stress and shear rate calculations are based. A similar effect is produced when the food is self-lubricating through oil exudation, for example, peanut butter. Slippage is also found in other commonly used shear-based tests such as the Couette, cone and plate, and parallel plates systems. Either way, slip occurs and the flow pattern is sufficiently altered, so that the application of equations developed for a perfect shear flow gives distorted and unrepresentative results. In many cases the occurrence of slip is self-evident. (A low flow index, on the order of 0.3 let's say, is almost certainly an indication of slip.) The degree of slip is usually unknown. It can be estimated by special experimental procedures, which involve re-

peating the measurements with sensors of different geometries (Yoshimura and Prud'homme, 1988; Wise et al., 2000). They are, however, quite elaborate and hence judged impractical. Consequently, reports on and discussions of slip in foods are rather rare in the food literature (Kokini, 1992; Ma and Barbosa-Canovas, 1995a,b).

The topic of this review, squeezing flow viscometry, has its own limitations (see below), but it offers a practical way to avoid the two abovementioned problems at least in principle. The following is a description of the different variants of the method, and a discussion of its potential applications in food research and quality assurance. The discussion focuses on foods such as tomato and bean pastes, yogurt, mustard, and mayonnaise. If they have any degrees of elasticity it plays only a minor role in the interpretation of their rheology. Materials such as wheat doughs whose rheology is dominated by elastic effects are not discussed in this work

II. CLASSIFICATION

Squeezing flow viscometry has several varieties. All share one common feature: the specimen is compressed vertically to induce a horizontal flow. A schematic view of the most common test geometries is given in Figure 1. They can be divided into two major categories:

1. Specimens with a constant volume and changing area (Figure 1, top).
2. Specimens with a constant area and changing volume (Figure 1, bottom).

The tests themselves can also be divided into two major types:

1. Application of a constant or other controlled displacement rate and monitoring the force vs. time or force vs. height relationship (Figure 1, left).
2. Application of a constant load and monitoring the height vs. time relationship (creep) (Figure 1, center).

The first kind of test is usually performed with a standard Universal Testing Machine. The instrument produces a constant displacement rate, and the resulting force is monitored by a load cell. The second kind is usually performed with a custom-made creep tester, where the displacement is monitored with a Linear Voltage Displacement Transducer (LVDT) or a micrometer.

The geometries shown in Figure 1 are particularly convenient for viscometry because symmetry considerations greatly facilitate the mathematical analysis of the tests results (see below). Squeezing flow however can be also induced in other geometries. The most notable are the one where one of the flat plates is replaced by a plate with an upward concavity (Meeten, 2001) and the other where the flat plates are not parallel and form a wedge (Chen, 1993). They are shown schematically in Figure 2. A variant of the two parallel plates array, especially developed for foods, is what has been called an 'Imperfect Squeezing Flow Array' (Figure 3), where the bottom plate is replaced by a shallow container (Lee and Peleg, 1992). This arrangement allows for the specimen to be formed or set in the detached container and then tested completely intact (see below).

The flow pattern during the fluid squeezing, can also be classified as being 'frictional' or 'lubricated'. The first is induced when there is a good contact, between the squeezed specimen and the sensor's plates through friction or bonding. The second type of flow is induced when the plates are lubricated, intentionally or by the specimen itself (slip). The pattern of the lubricated squeezing flow is also known as a 'plug flow'. The forces that evolve during a plug flow are considerably lower than those when there is friction between the fluid and plates (see below). The two flow patterns can be discerned visually from the shape of the exiting fluid's front. It has a parabolic profile in frictional flow, and a flat profile in lubricated flow (Figure 4).

III. THEORY

The specimen in squeezing flow viscometry must have a large aspect ratio (diameter to height

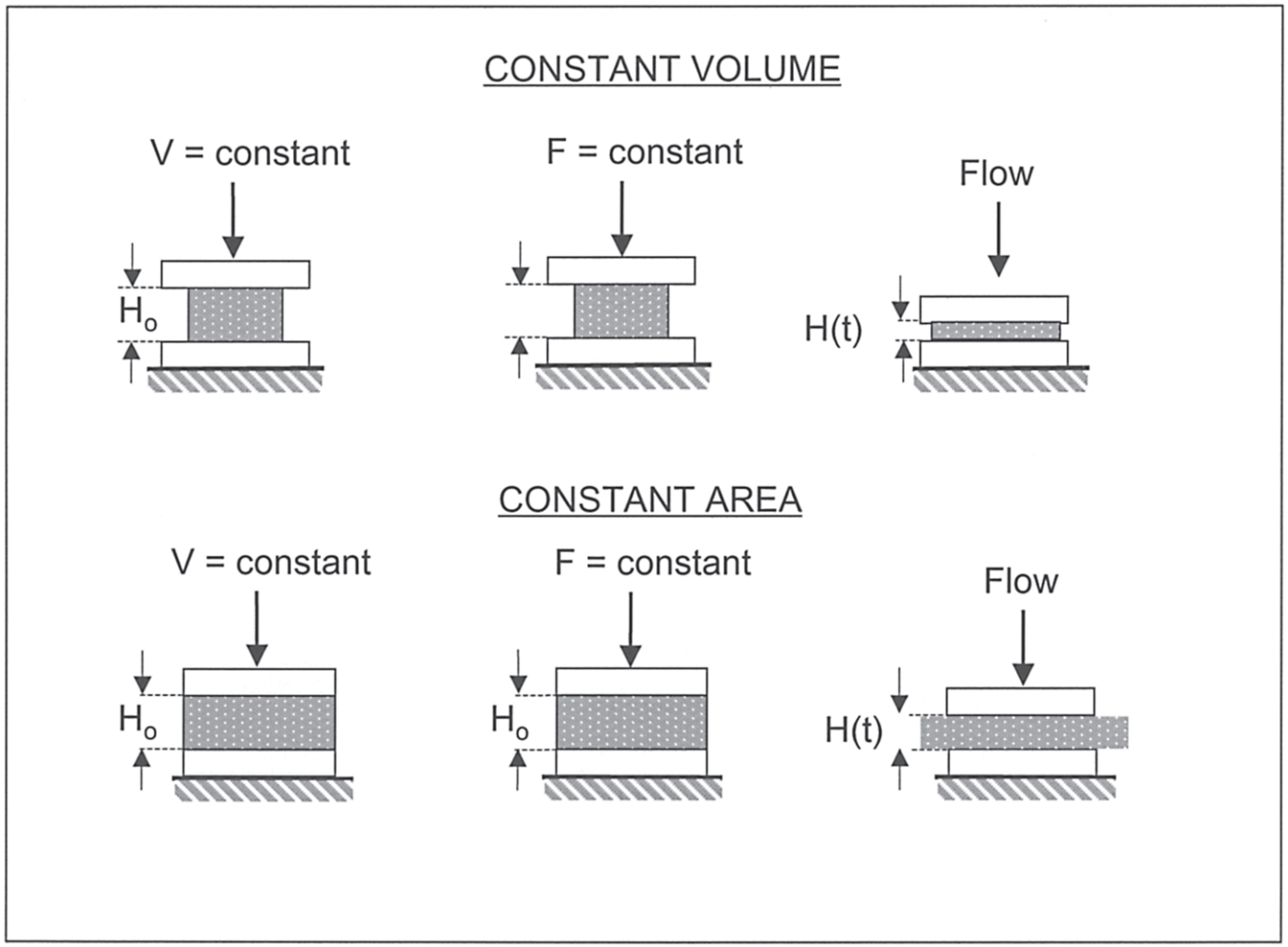


FIGURE 1. The basic test configurations of squeezing flow viscometry with parallel plates as a sensor.

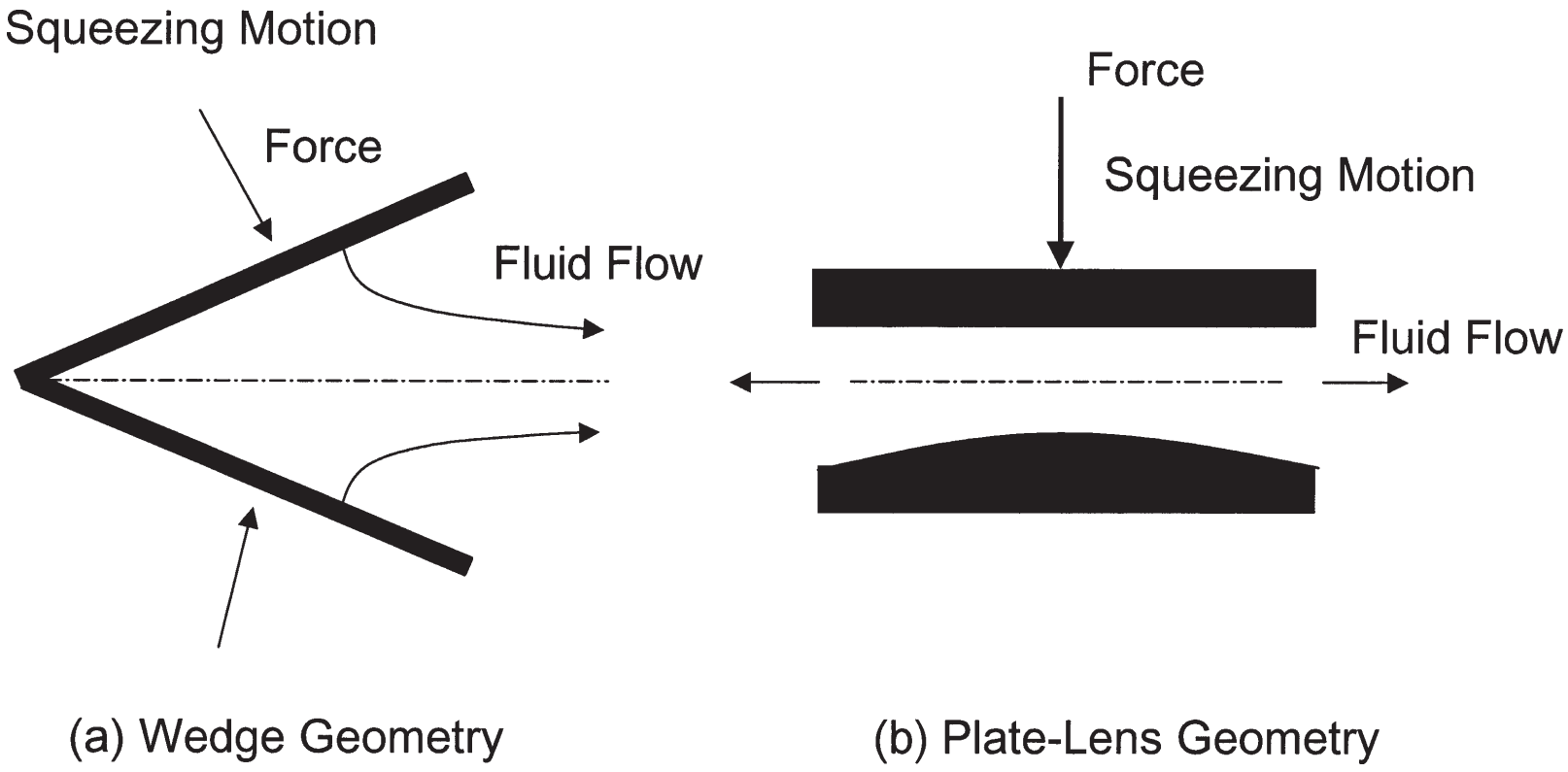


FIGURE 2. Wedge and plate-lens geometries, which produce squeezing flow.

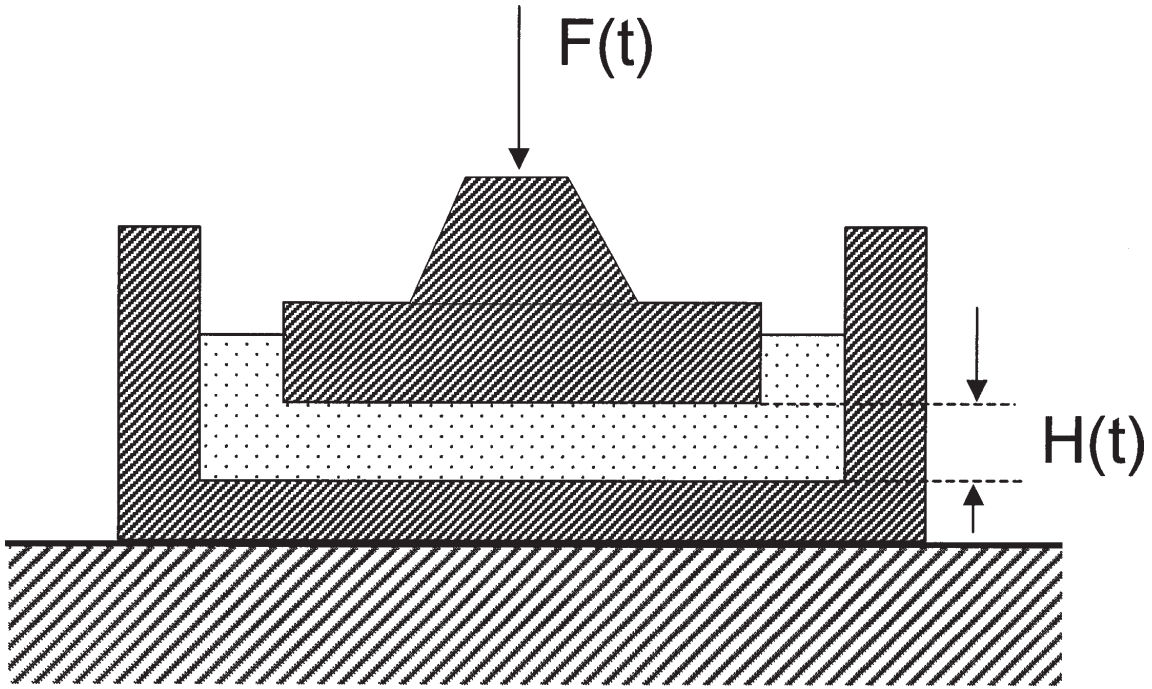
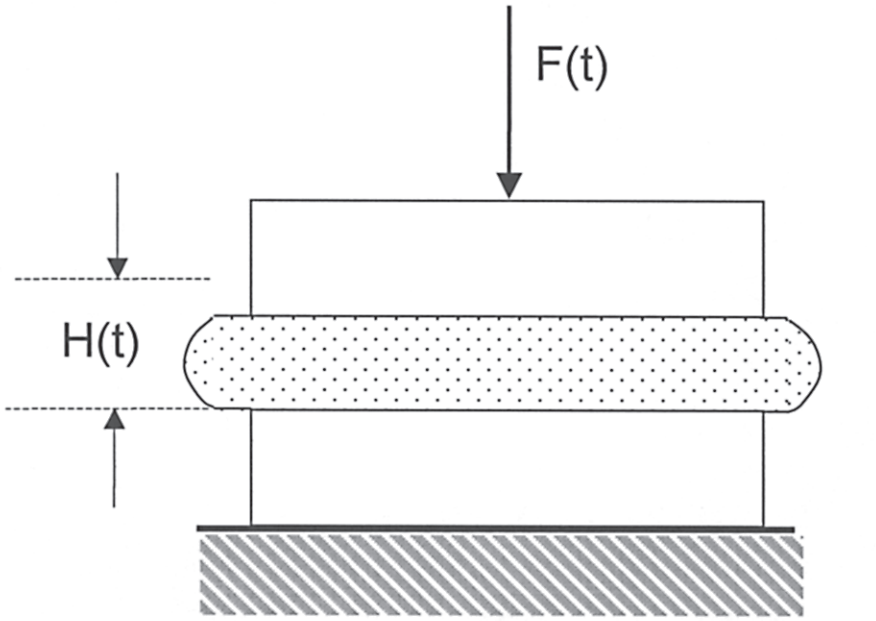
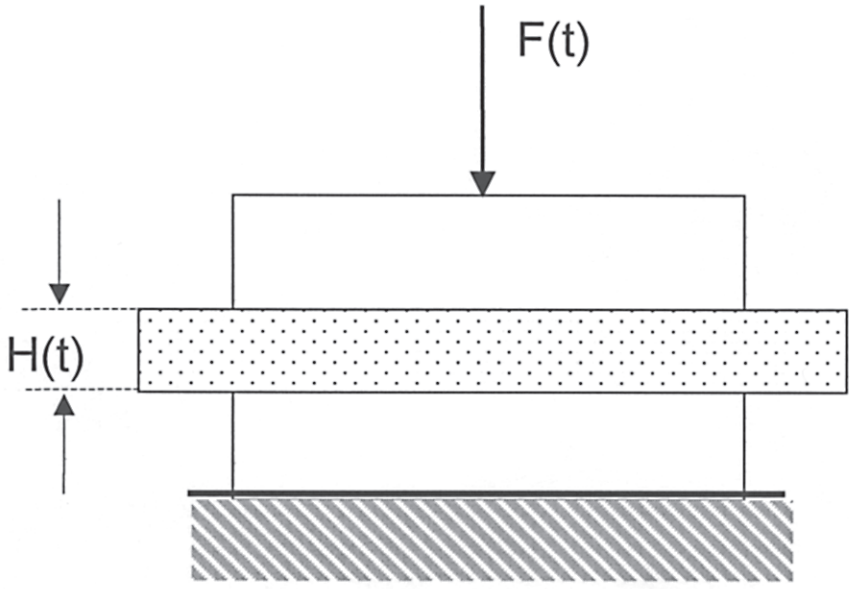


FIGURE 3. An ‘imperfect’ squeezing flow sensor where the bottom plate is replaced by a shallow container.



(a) Frictional Squeezing Flow



(b) Lubricated Squeezing Flow

FIGURE 4. Schematic view of the exiting fluid profile in frictional and lubricated squeezing flow between parallel plates.

ratio), on the order of 10 to 30. There are two reasons for this requirement: (1) to increase the test's sensitivity (see below) and (2) to reduce the influence of end effects. The aspect ratio's upper limit is usually determined by practical considerations: the dimensions of the testing machine on which the sensor is mounted, and the ability to guarantee parallel surfaces (see below). Because the more commonly used sensor in squeezing flow viscometry consists of two parallel plates (Figure 1), the following will focus on this particular geometric array. Theoretical analyses of the flow patterns in other geometries can be found in the literature (e.g., Chen, 1993; Wilson, 1993; Meeten, 2001). It is unlikely that they will have a major impact on food viscometry anytime soon and therefore will not be discussed further. The geometry of squeezing flow between parallel plates and its corresponding nomenclature is shown in Figure 5. We will use H or H(t) to symbolize the momentary specimen height, that is, the distance separating the plates, and R or R(t) the plates or specimen momentary diameter as the case might be. The reader will notice that in many publications, especially the early ones (e.g., Leider and Bird, 1974a,b; Sherwood and Durban, 1996), half the height or, h, is used in the equations because of symmetry considerations. Because what is actually monitored in the most common test, where V = constant and F variable, is the total distance between the plates H(t), we will use it exclusively because others have also done (e.g., Avila and Binding, 1982). The force-height-time relationship in squeezing flow, as one would expect, depends on both sensor geometry and test conditions (e.g., a constant load or constant displacement rate) and on the rheological properties of the tested fluid (e.g., Newtonian, pseudoplastic, Hershel-Buckley, etc.). In addition, it will also depend on whether there is enough lubrication to produce a plug flow or strong enough contact to produce a fully developed frictional flow. The following describes the theoretical aspects of the most commonly encountered squeezing flow regimes. Departures from the ideal and the effect of measurement artifacts are discussed separately later. Because of the ease to form a specimen with controlled dimensions (see below), the most practical sensor for squeezing flow viscometry for soft foods is the one based on a constant area and

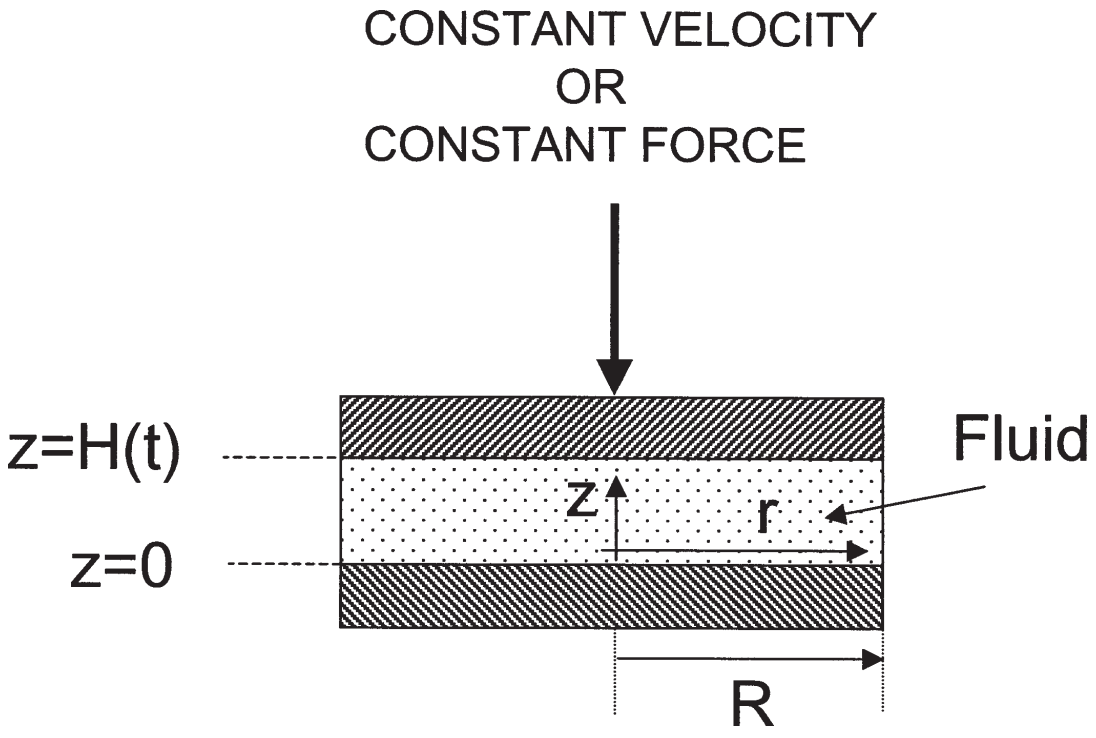


FIGURE 5. The geometry of squeezing flow between two parallel plates of equal diameter.

changing volume (Figures 1, 4, and 5). Thus, unless otherwise stated, all the equations will refer to this kind of an experimental array. (The constant volume and changing area configuration is more convenient for testing 'hard' materials, such as certain polymers. Discs prepared from such materials do not significantly deform when handled prior to testing.)

A. Frictional (Shear) Flow

When there is sufficient friction between the fluid and the plates, one can assume that the horizontal velocity of the liquid in contact with the plates is zero, and elsewhere a function of the distance from the plates only. Because at the center the fluid does have a finite horizontal velocity higher than zero, the result is an inevitable shear, or, in other words, in all nonlubricated squeezing flows the fluid is both elongated and sheared.

The flow of a Newtonian fluid between two parallel plates is calculated by the Stefan's equation (Leider and Bird, 1974a):

$$F(t)=\frac{3\pi R^4(t)\mu}{2H^3(t)}\left(-\frac{dH(t)}{dt}\right) \tag{1}$$

where F(t) is the momentary force, H(t) the momentary specimen's height, R(t) the plate's radius and μ the fluid's shear viscosity.

For a constant displacement flow, –dH(t)/dt=V, and therefore when the area is constant, R(t) = R, Eq. 1 becomes:

$$F(t)=\frac{3\pi R^4\mu V}{2H^3(t)} \tag{2}$$

In the creep array if both the load and the area are constant, that is, F(t)=W and R(t)=R, integration of Eq.1 yields:

$$H(t)=H_0\sqrt{\frac{1}{1+\frac{4H_0^2Wt}{3\pi\mu R^4}}} \tag{3}$$

where H_0 is the specimen's initial height.

B. Non-Newtonian Fluids

The constitutive equation of a pseudoplastic fluid, which takes into account the shear and extensional components of the flow simultaneously, can be expressed in the tensorial form as:

$$\underline{\underline{\tau}} = K\left(\underline{\underline{\dot\gamma}}:\underline{\underline{\dot\gamma}}\right)^{\frac{n-1}{2}}\underline{\underline{\dot\gamma}} \tag{4}$$

where $\underline{\underline{\tau}}$ and $\underline{\underline{\dot\gamma}}$ are the shear stress and strain rate tensors, respectively, K the consistency coefficient, and n the flow index. The equivalents of Eqs. 2 and 3 and (Campanella, 1987) for this case are

1. For a test performed at constant displacement rate -dH(t)/dt=V, and a constant area, R(t)=R:

$$F(t)=\frac{2\pi K R^{n+3}}{n+3}\left(\frac{2n+1}{n}\right)^n\frac{V^n}{H(t)^{2n+1}} \tag{5}$$

2. For a creep test under constant stress, F(t)=W:

$$\frac{1}{H(t)^{\frac{n+1}{n}}}=\frac{1}{H_0^{\frac{n+1}{n}}}+\frac{n+1}{2n+1}\left(\frac{W(n+3)}{2\pi KR^{n+3}}\right)^{\frac{1}{n}}t \tag{6}$$

The situation becomes more complicated when the tested fluid has a substantial yield stress (τ_0), in which case a shear free zone is formed in the central region between the plates (Campanella and Peleg, 1987a; Meeten, 2000). It should be mentioned that the concept that fluids have a true yield stress has been challenged (Barnes and Walters, 1985; Barnes, 1992). The argument has been that given enough time flow would be eventually detected. Because most foods are biologically, chemically, or physically unstable, long-term experiments to determine their yield stress are impossible in principle. Therefore, one can only relate to an "apparent yield stress", which dictates the food rheological behavior on the pertinent time scale. With this in mind one can

determine its magnitude from the asymptotic height, H_A, in a creep test under a constant stress after a long time is elapsed (Campanella and Peleg, 1987a) as shown in Figure 6, that is,

$$\tau_0 = \frac{3WH_A}{2\pi R^3} \tag{7}$$

Theoretically, this equation implies that the asymptotic thickness of a fluid without a yield stress will always be zero, and proportional to the yield stress when it exists, irrespective of the specimen's initial height.

In principle, the Newtonian viscosity μ or the consistency coefficient, *K*, and the flow index, *n*, of a food can be determined from their plots of log F(t) vs. log H(t) relationship in constant displacement tests or log H(t) vs. t relationship in a creep experiment. No doubt these analyses would be the preferable options had slip not been a problem. This is because the forces that develop during frictional flow, theoretically, can be orders of magnitude higher than those in lubricated squeezing flow, depending on the aspect ratio. Thus, if adequate friction could be guaranteed, the test's sensitivity would increase dramatically (see below).

One should also remember that Newtonian liquids, such as honey or maple syrup, do not exhibit slip. Hence, if squeezing flow viscometry is applied to such liquids (in the imperfect array, for example, see below) their shear viscosity, μ, can be estimated using Eqs. 2 or 3, depending on whether the displacement rate or the load remains constant during the test. If in a constant displacement rate experiment the absolute slope of the log F(t) vs. log H(t) plot is smaller than 1.0, one can safely conclude that the flow is lubricated and not frictional. However, if the absolute slope is anywhere between 1.0 and 3.0, it is uncertain whether the specimen is pseudoplastic or self-lubricating (Hoffner et al., 1998). In principle it can be considered plastic if the slope is only very slightly above 1.0 (see below).

C. Lubricated Squeezing Flow

The differential equation that governs the flow of a Newtonian fluid squeezed between frictionless parallel plates in a constant area configuration is (Chatraei et al., 1981, Soskey and Winter, 1985)

$$F(t) = \frac{3\pi\mu R^2}{H(t)}\left(-\frac{dH(t)}{dt}\right) \tag{8}$$

Thus for the case of a constant displacement rate (- dH(t)/dt = V) the force–height relationship is given by:

$$F(t) = 3\pi\mu R^2 \frac{V}{H(t)} \tag{9}$$

The reader should be reminded that Newtonian fluids by themselves do not slip, and therefore Eqs. 8 or 9 are only applicable in situations where the plates are deliberately lubricated with a fluid having a negligible viscosity. The difference between Eq. 8 and Eq. 1, apart from the factor of 2, is that in the former F(t) is proportional to [R/H(t)][-dH(t)/dt], while in the latter to [(R/H(t))^3][-dH(t)/dt]. Because we are dealing with specimens having a very large aspect ratio (large diameter and small height) this difference dictates that under the same test conditions, that is, the same sensor geometry and displacement rate, lubrication of the plates will cause a dramatic decrease of the forces. Thus, in order to generate forces of a similar magnitude, the plates in lubricated squeezing flow viscometry must be considerably wider than in frictional flow viscometry.

It can be argued that in most Non-Newtonian foods, it is easier to produce a displacement pattern that is closer to a frictionless squeezing flow than to guarantee the type of contact with the plates needed for a fully developed shear (frictional) flow. Hence, the lubricated squeezing flow option seems to be preferable in food viscometry despite the need to employ a wider sensor. As shown below, a sensor with plates having a diameter on the order of 10 cm mounted on commonly available testing machines is more than sufficiently sensitive to test almost every semiliquid food. The assumption that the contact between the liquid and plates can be treated as practically frictionless, as already mentioned, can be verified by inspecting the slope of the log F(t) vs. log H(t) relationship (see below).

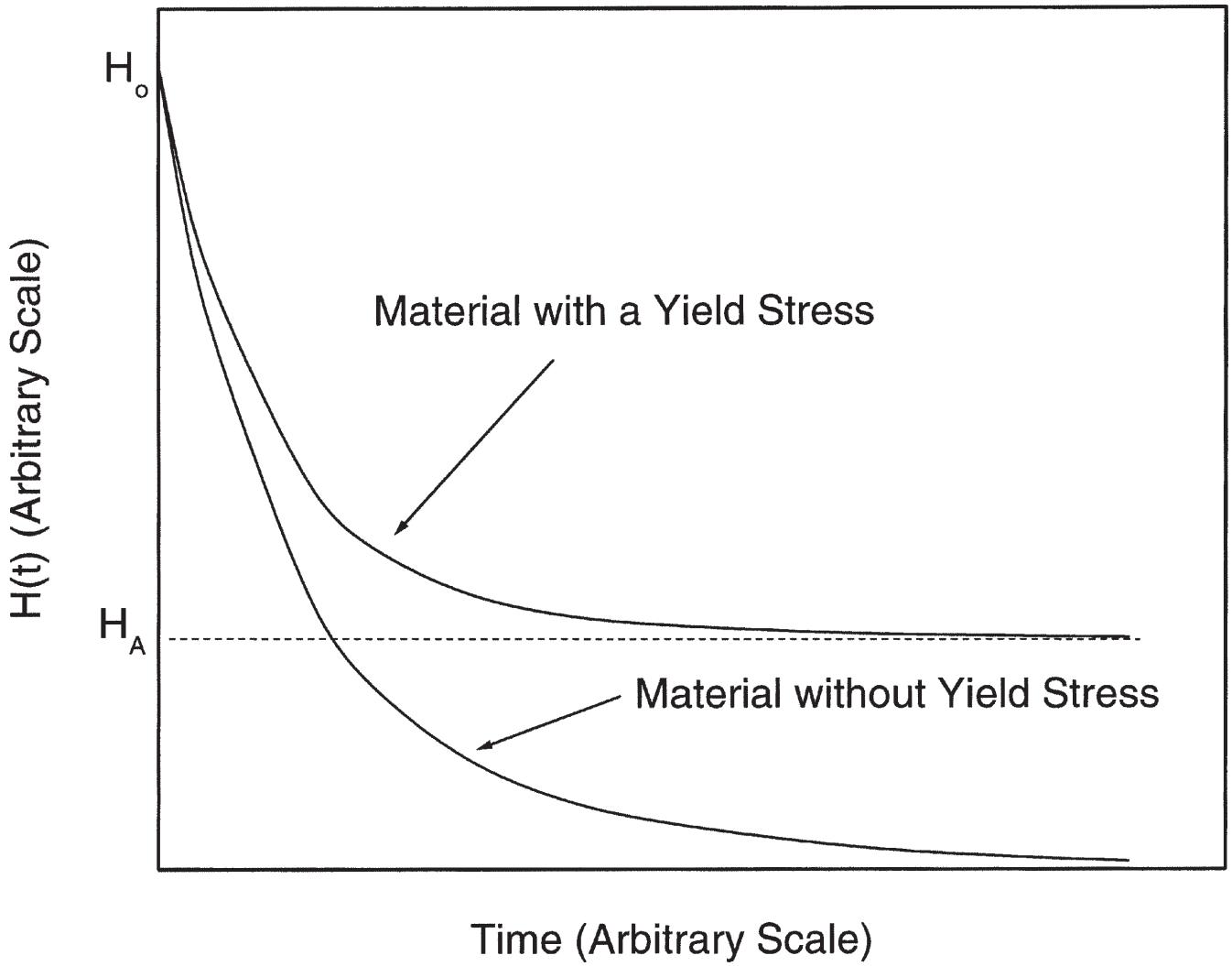


FIGURE 6. Schematic view of the creep curves of a fluid with and without a yield stress.

In a constant stress (creep) test, under a load W the flow of a Newtonian liquid is governed by Eq. 8, which becomes:

$$H(t) = H_0 \, e^{-\frac{W t}{3 \pi R^2 \mu}} \tag{10}$$

Eq. 10 entails that the specimen's height diminishes exponentially. (When there is adequate friction between the fluid and plates, the height's decrease is governed by Eq. 3.)

For a pseudoplastic fluid with a consistency coefficient K and flow index n the corresponding flow equations are (Campanella, 1987; Campanella and Peleg, 1987b; Lee and Peleg, 1992):

$$F(t) = 3^{\frac{n+1}{2}} K \pi R^2 \left(\frac{V}{H(t)} \right)^n \tag{11}$$

and

$$H(t) = H_o \, e^{-\left(\frac{W}{3^{\frac{n+1}{2}} K \pi R^2} \right)^{1/n}} \tag{12}$$

In lubricated squeezing flow, the absolute slope of the log F(t) vs. log H(t) relationship at a constant displacement rate (Eq. 11), as already mentioned, must be smaller than one, when compared with between 1.0 and 3.0 in frictional flow. In creep tests under a constant load (Eq. 12), a plot of log H(t) vs. t will also yield a linear relationship. Consequently, there are situations where the distinction between lubricated and frictional is very simple regardless of the test's type. (It has been suggested to use the slope of the log F(t) vs. log H(t) relationship to test whether changing a coaxial viscometer's surface finish by grooving, for example, indeed would eliminate slip [Hoffner et al., 1998].) However, again, if the absolute slope of the log F(t) vs. log H(t) relationship is smaller than one, then it is almost certain that the flow is practically frictionless. However, if the absolute slope is in the range 1 to 3, the possibility that partial slip occurs cannot be ruled out.

D. Elongational Viscosity

Lubricated squeezing flow produces what is known as elongational or biaxial flow. It is so called because the squeezed fluid stretches radially and azimuthally as it flows outward. This kind of flow is regulated by what is known as the "elongational viscosity" μ_b. It is defined as the ratio between the applied normal stress, $F(t)/(\pi R^2)$, and the biaxial strain rate, $d\varepsilon_b/dt = V/[2H(t)]$ for the case of a constant displacement rate $-dH(t)/dt = V$, that is,

$$\mu_b = \frac{2 F(t) H(t)}{\pi R^2 V} \tag{13}$$

The elongational viscosity, μ_b, is determined directly from the experimental force-height relationship, the plate radius and the displacement rate, and no model is required for its calculation. The reader will notice that because of the specimen's progressively diminishing height, a constant displacement rate produces a continuously increasing biaxial strain rate. Hence, meaningful comparison of the extensional viscosity of Non-Newtonian fluids, like their shear viscosity, must be done a comparable rate.

Ideally, the extensional or elongational viscosity of a Newtonian fluid, μ_b, is given by:

$$\mu_b = 6 \mu \tag{14}$$

where μ is the shear viscosity, likewise it is rate independent. In contrast a pseudoplastic fluid does have a strain rate-dependent elongational viscosity, which is given by:

$$\mu_b = 3^{\frac{n+1}{2}} \, 2^n \, K \, \dot{\varepsilon}_b^{\,n-1} \tag{15}$$

A schematic view of the μ_b vs. $\dot{\varepsilon}_b$ plot for Newtonian and pseudoplastic fluids is shown in Figure 7. It could be noted that for $n = 1$ and $K = \mu$, that is, Newtonian fluids, Eq. 15 simplifies to Eq. 14. Theoretically, the slope of the double logaritmic plot of a pseudoplastic liquid would be n-1 and independent of the displacement rate, V. The theoretical limiting cases are an ideal

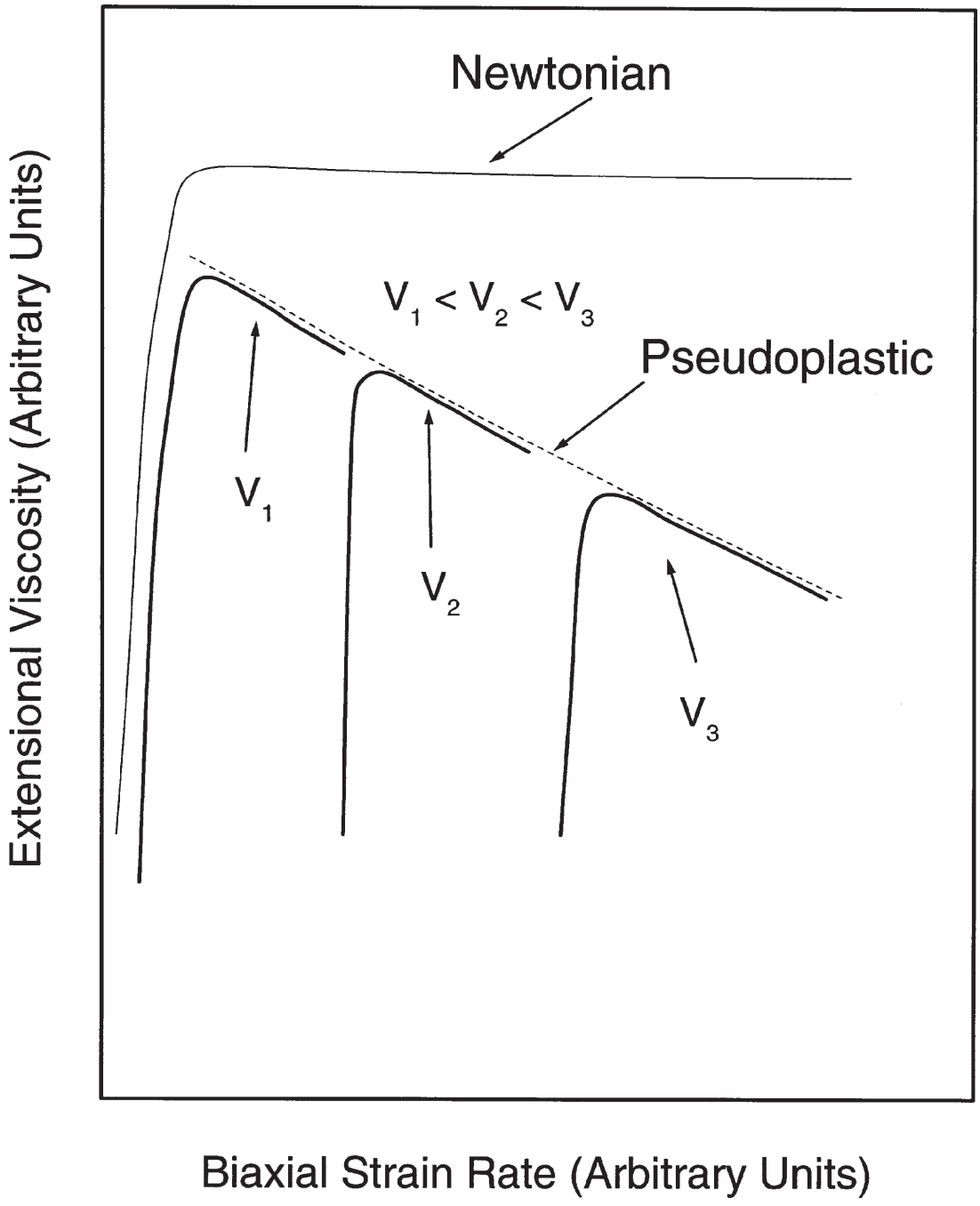


FIGURE 7. Theoretical elongational viscosity vs. biaxial strain rate relationships of ideal newtonian and pseudoplastic fluids determined by squeezing flow viscometry at different displacement rates (V1, V2, and V3).

Newtonian liquid where the slope is zero (rate independent μ_b) and an ideal plastic fluid where the slope is –1 (see below).

1. Stress Relaxation

Theoretically, nonelastic fluids that do not have a yield stress cannot sustain shear or normal stresses. Consequently, when the flow ceases that is the motion of the testing machines crosshead is halted, the force ought to drop to zero instantaneously. In reality some time must elapse before the force totally dissipates, a consequence of the fact that real instruments have a finite non-zero response time and that the crosshead ought to decelerate before it comes to a full stop. These factors, however, have only a minor influence on the recorded force decay rate. They should have no effect at all on the residual force after relaxation. Thus, if the force does not relax almost instantaneously and/or does not completely dissipate during a period of 2 to 3 min, let's say, then one must conclude that the tested specimen has either a certain degree of elasticity, a considerable yield stress or both (see below).

2. Imperfect Squeezing Flow

The imperfect squeezing flow (Figure 3) allows testing food specimens virtually intact. This would be accomplished if the specimen is formed in the shallow container or collected directly from the filling machine in an industrial operation and then transferred to the laboratory for testing. Maintaining the sample's integrity comes at the cost of loosing accuracy and sensitivity, caused by the more complicated test geometry, the upward annular flow in the sensor and the creation of buoyancy forces. The role of these forces would obviously be more significant in lubricated flow where the squeezing forces are generally of a much smaller magnitude than in frictional flow (Damrau and Peleg, 1997). To reduce the impact of these artifacts one must use the widest sensor deemed practical, operate at high compression ratio (i.e., reach very small specimen heights) and maintain a wide gap between the upper plate and the container's wall. The efficacy of these measurements can be assessed by testing the fluid with sensors of different diameters and gaps. Again, it has been shown that reasonable results can be obtained with sensors having an upper plate diameter on the order of 10 cm and gaps of about 2 cm or less (Hoffner et al., 1997). It ought to be added that if a stress relaxation test is performed with the "imperfect" array, the force of an ideal liquid will drop not to zero but to the buoyancy force. A simple calculation shows that for most foods tested with sensors of the above-mentioned geometry and size, whose density is on the order of 1 g/cm^3, this would translate to a buoyancy force of about 0.5 N per mm of immersion. Therefore, for viscous semiliquid foods such as mayonnaise and ketchup, not to mention bean paste, the buoyancy force has only a minor effect on the overall force levels, and on the residual force after relaxation. In many cases the magnitude of the buoyancy forces is smaller than the variation among replicates (see below). Although clearly an artifact and a source of a consistent error, buoyancy can usually be considered a negligible factor when the test's results are interpreted.

IV. FOOD APPLICATIONS

Dr. Edward B. Bagley and his collaborators at the USDA-NRRC (Peoria, IL) introduced squeezing flow viscometry to food research in the mid-1980s (Casiraghi et al., 1985). Since then, it has been applied to a growing number of foods, among them:

- Butter (Shukla et al., 1995; Shukla and Rizvi, 1997).
- Cheeses:
  - Processed American (Campanella et al., 1987c).
  - Mozzarella (Casiraghi et al., 1985; Ak and Gunasekaran, 1996; Wang et al., 1998)
  - Ricotta (Suwonsichon and Peleg 1999b).
- Corn meal/masa (Ramirez-Wang et al., 1996; Wong et al., 1996; Lo et al., 1999; Limanond et al., 1999).
- Honey (Damrau and Peleg 1997).

- Ketchup (Campanella and Peleg 1987b, Lee and Peleg 1992, Lorenzo et al., 1997)
- Maple syrup (Damrau and Peleg 1997).
- Mayonnaise (Campanella and Peleg 1987b, Hoffner et al., 1997, Corradini et al., 2000a,b).
- Milk sweet ("Dulce de leche") (Corradini and Peleg, 2000).
- Mustard (Hoffner et al., 1997, 1998, Suwonsichon and Peleg, 1998; Corradini et al., 2000a).
- Peanut butter (Campanella and Peleg, 1987a).
- Refried beans (Suwonsichon and Peleg, 1999a).
- Tomato paste (Lee and Peleg 1992, Lorenzo et al., 1997, Corradini et al., 2000a,b).
- Wheat dough (Huang and Kokini, 1993; Battacharya et al., 1999; Wilkstrom and Bohlin, 1999).
- Yogurt (Suwonsichon and Peleg, 1999c; Corradini et al., 2000b).

The parameters reported were the consistency coefficient (K) and flow index (n), the apparent yield stress (τ_0), the apparent stress at a given height or after relaxation for a given time (Table 1) and the elongational viscosity (μ_b) as a function of the biaxial strain rate (Table 2). The results shown in the tables were obtained with the parallel plates geometry or in the "imperfect" array, that is, where the bottom plate had been replaced by a shallow container. It has been

TABLE 1
Apparent Stresses of Various Semiliquid Foods During Squeezing Flow between Parallel Teflon or Teflon-Coated Plates (Determined at 1 mm Height and a Displacement Rate of 1 to 6 mms^{-1}

Food	Apparent Stress at 1mm (kPa)	Apparent Stress After Relaxation for 120s (kPa)	Reference
Mayonnaise Almost intact Stirred	 ~10 ~8	 3-4 ~2-5	Corradini et al., 2000a
Mustard Without seeds With seeds	 2-4 2-4	 ~1 1-2.5	Corradini et al., 2000a Suwonsichon and Peleg, 1998
Milk sweet	18-40	4-10	Corradini and Peleg, 2000
Refried beans (at 1.5mm)	9-26	3-15	Suwonsichon and Peleg, 1999a
Ricotta Cheese Whole milk Skim milk Fat free	 5-12 8-14 11-18	 1-6 4-7 1-8	Suwonsichon and Peleg, 1999b
Tomato paste Almost intact Stirred	 9-16 ~13	 2.5-5 ~3.5	Corradini et al., 2000a,b
Yogurt Almost intact Stirred	 3-5 0.2-1	 1-3 0.1-0.4	Suwonsichon and Peleg, 1999c

TABLE 2
Elongational Viscosity (μ_b) of Various Foods Determined by Lubricated Squeezing Flow Viscometry (Approximate Rounded Values)

Food	$\dot{\varepsilon}_b$ (s^{-1})	Temperature (^oC)	μ_b $(kPas)$	Reference
American Cheese	0.015	45 62	120-200 40-60	Campanella and Peleg, 1987c
Butter (From anhydrous milk fat)	0.025	17 22 27	10500 3500 350	Shukla et al., 1995; Shukla and Rizvi, 1997
Corn/masa	0.01 0.001	ambient	5500 4000	Ramirez-Wong et al., 1996
Ketchup	0.1	ambient	20-25	Lorenzo et al.,
Mayonnaise	0.1	ambient	50-70	Hoffner et al., 1997
Mozzarella Cheese	0.015 0.15	40 60 40 60	550 250 90 30	Ak and Gunasekaran, 1996
Mustard	0.1	ambient	~30	Hoffner et al., 1997
Tomato Paste	0.1	ambient	100-150	Lorenzo et al., 1997

shown that the rheological parameters obtained by squeezing flow viscometry are highly reproducible that is on the order of 5% or less (e.g., Hoffner et al., 1997; Corradini et al., 2000a,b; Suwonsichon and Peleg, 1998, 1999a,b,c). The differences between food products of different brands or of the same brand produced on different dates are frequently considerably higher, and they can easily be detected by the method irrespective of the array employed (Lorenzo et al., 1997; Suwonsichon and Peleg, 1998, 1999a,b,c).

It has also been shown that if the specimen is gently transferred from its container onto the bottom plate of the imperfect squeezing flow array using a wide spoon or spatula, it suffers very little damage and hence can be tested practically intact (Suwonsichon and Peleg, 1999c). This has been demonstrated by comparison of the measurements scatter with that of three mineral oils. The oils have no internal structure to be destroyed and hence they do not suffer any damage during handling. Consequently, the scatter in these measured rheological parameters is a measure of the test's reproducibility (Corradini et al., 2000a — see below). This may not be the case if the specimen is very sticky and cannot be easily removed from the spoon or spatula (e.g., peanut butter). The potential implication of stickiness is yet to be studied.

Another major advantage of the squeezing flow viscometry over coaxial and capillary methods is that it allows testing of foods that contain particulates. Mustard with seeds and refried beans are two examples (Suwonsichon and Peleg, 1998, 1999a). As long as the suspended particles are not larger than the specimen's final height, the resulting force-height relationships remain smooth and can be interpreted in the usual manner.

A. Effect of the Initial Specimen Height

Unless the specimen is formed in the sensor, it is difficult if not impossible to guarantee that its height will be uniform and controlled. Thus, specimens transferred onto the bottom plate with a wide spoon or spatula would inevitably be of variable and uneven thickness. This, however, seems not to be a significant problem in food testing. The reason is that the relevant data for the rheological parameters calculation are in the force-height relationship where the specimen height has been reduced considerably, that is, when R/H(t) is large numerically. Thus, the initial height variations primarily affect the transient flow regime, which is not taken into account in the results interpretation (Figure 8). The reader will also notice that the initial height (H_o) does not appear in the flow equations except those that describe the creep behavior (see Eqs. 3, 6, 10, and 12). One can also argue that had the variation in the specimen's initial height been a significant factor the test's reproducibility would be much lower than that actually observed.

B. Rates and Their Effects

Had all semiliquid foods been truly pseudoplastic fluids, the rate effects should have been predicted by Eq. 5. There is evidence that this is not the case in several products, such as mayonnaise, mustard, and tomato paste (Corradini et al., 2001). The disagreement cannot be explained as being caused just by slight deviations from pseudoplasticity or by viscoelasticity. The magnitude of the discrepancy suggests that these foods have partial solidity, a characteristic consistent with the considerable residual stress observed after relaxation of the stress (see Table 1). Therefore, the rate effect can be assessed in the following manner. Let us assume that the stress has two major components, one, the residual stress after-relaxation that represents the specimen's solidity and hence is rate independent, and the other the dissipating stress, which is rate dependent. The "solid

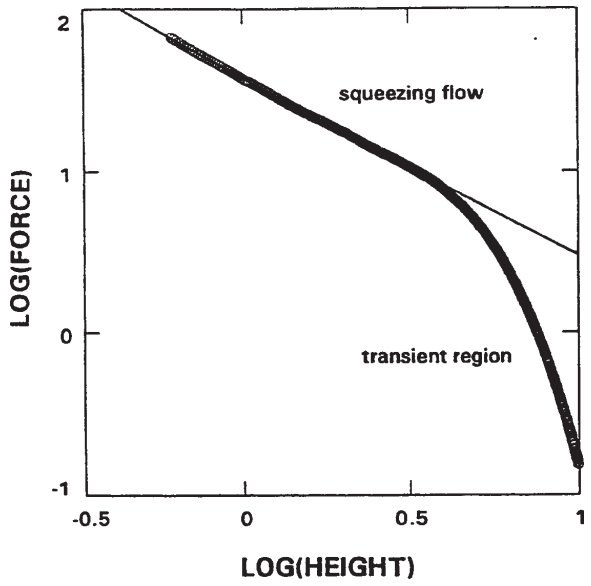


FIGURE 8. Typical experimental log force-log height relationship in squeezing flow viscometry between parallel plates of equal diameter.

part" is not an elastic component in the usual sense, that is, its magnitude is not proportional to the displacement. It represents the ability of the deformed structure to maintain or recover its integrity after flow and is therefore reminiscent of yield stress of a sheared liquid, which is observed after the shearing ceases.

According to this oversimplified model, the relation between forces recorded at the same height at two different speeds V_1 and V_2, can be approximated by the empirical relationship (Corradini et al., 2000):

$$\frac{F_{V2} - F_R}{F_{V1} - F_R} = \left(\frac{V_2}{V_1}\right)^m \tag{16}$$

where F_{V2} and F_{V1} are the forces measured at speeds V_1 and V_2, respectively, F_R is the residual unrelaxed force at the corresponding height, assumed to be roughly constant, and m is a constant. Special cases are a Newtonian liquid where $F_R = 0$ and $m = 1$ and hence $F_{V2}/F_{V1} = V_2/V_1$, and an ideal pseudoplastic fluid where $F_R = 0$ and $m = n$, the flow index, and hence $F_{V2}/F_{V1} = (V_2/V_1)^n$.

The value of F_R can be directly determined from experimental relaxation curves as that illustrated in Figure 9. As shown by Suwonsichon and Peleg 1999a,b,c; Corradini and Peleg, 2000, and Corradini et al., 2000a,b, in almost all cases it indeed remained fairly constant at the range of the speeds tried. If Eq.16 indeed captures the rate effect then the magnitude of m should be independent of the speeds ratio. This can be verified by comparing the magnitudes of the calculated m values from tests performed at different compression rates. The value of m can be conveniently calculated from:

$$m = \frac{\log \dfrac{F_{V2} - F_R}{F_{V1} - F_R}}{\log \dfrac{V_2}{V_1}} \tag{17}$$

Values of m for mayonnaise, mustard, and tomato paste determined at four speeds ratios,

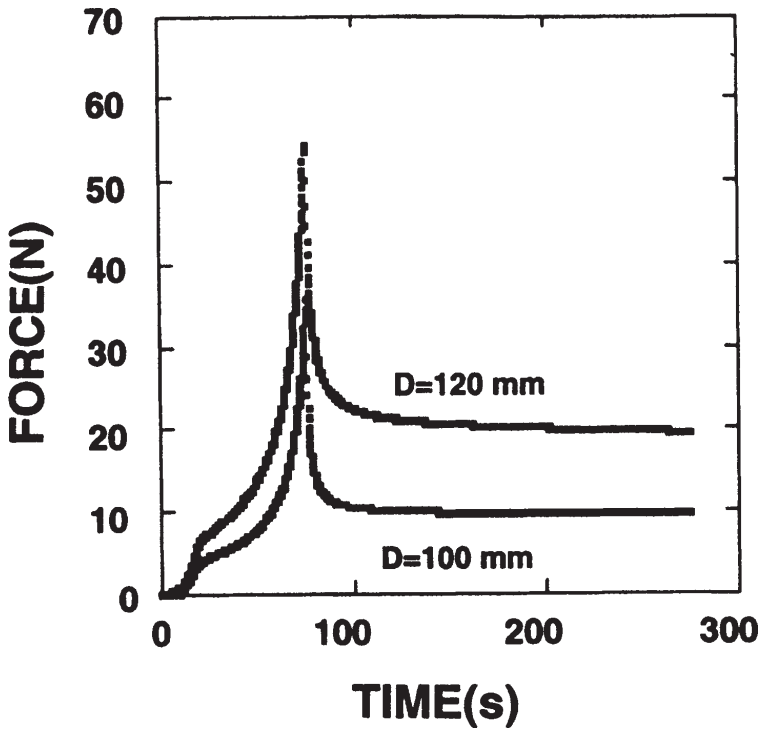


FIGURE 9. Typical experimental force-time (displacement and stress relaxation), in squeezing flow viscometry between parallel plates of equal diameter. Results for plates with diameters 100 mm and 120 mm are shown.

corresponding to speeds 5, 10, 15, 10, and 25 mm/min, were reported by Corradini et al. (2000a). They were all fairly constant, that is, practically independent of the compression rate, but characteristic of the particular product or brand. The *m* values were all on the order of 0.15 to 0.33, that is, of a magnitude well below that of the characteristic flow index of a typical pseudoplastic fluid, which is expected to be at least about 0.6 if not higher. This suggests that the flow pattern of at least some semiliquid foods may be governed by their plasticity. Support for this view came from comparison of these results with those of similar tests performed on the three silicone oils. (The tests were performed in an "imperfect array" so that the oils could be contained.) The coefficient of variation in the oil tests was on the same order of magnitude as that of the foods, that is, it rarely exceeded 10%, and in most cases was below 5%. Being Newtonian or approximately Newtonian (at least at low rates), the oils were not supposed to slip, and the stress at a given height therefore was expected to be proportional to the compression rate (i.e., $F_{V1}/F_{V2} = V_1/V_2$). This was indeed observed in all three oils and one must conclude that the observed foods behavior was not an instrumental artifact. (The very slight deviations from the theoretical values could be attributed to buoyancy, annular flow and end effects, which were created by replacing the bottom plate with the shallow container.)

The peculiar rate effects are also manifested in another way. Typical plots of calculated elongational viscosity vs. the biaxial strain rate of tomato paste and mayonnaise (Corradini et al., 2000a) are shown in Figure 10. They clearly demonstrate that the elongational viscosity calculated with Eq. 15 was not a unique function of the strain rate as would be expected from a Newtonian or pseudoplastic fluid (compare with Figure 7). Qualitatively, the observed behavior reflected is an intermediate flow pattern between that of pure plasticity and pseudoplasticity, which is consistent with the observed rate dependence of the apparent stresses (see Figure 10). However, because imperfect lubrication could also be a factor, at least theoretically, a direct relationship between the apparent elongational viscosity and the rate could not yet be established. It should also be added that had friction been a significant factor, a shear free deformation could no more be assumed and the calculation of μ_b using Eq. 15 would become meaningless.

V. ARTIFACTS

The accuracy of all rheological testing methods (or of any testing method for this matter) is never absolute. Even when the test is performed "correctly", in a properly calibrated instrument, errors are inevitable. In our case the errors are of two types, theoretical and instrumental.

A constitutive equation of a complex food material (like Eq. 4) is only an approximate model of the food's actual rheological behavior. If derived from a single type of tests, flow let's say, the model needs not always account for other types of rheological responses such as viscoelastic phenomena. Thus, parameters such as the consistency coefficient or flow index, which are calculated with an equation derived for a pseudoplastic fluid, can only be used to quantify the food properties if the food is indeed or at least approximately a pseudoplastic fluid. This may not be a problem if the purpose of the test is just to compare products, assess the role of formulation, or quantify the effects of handling, etc. However, the model can be ineffective if one tries to predict the behavior of semiliquid foods under conditions that differ considerably from those that existed in the tests used for its determination. The rate effect, which has been discussed earlier, is an illustrative example. As already mentioned, this is a more serious problem when conventional viscometric methods are used to assign the rheological properties of foods because the tested specimen is partially sheared before the test even begins.

The second type of error stems from imperfections, some inevitable, in the sensor's design and the instrument's construction.

A. Friction and Lubrication in Squeezing Flow Viscometry

The flow equations, from which rheological properties are derived, are based on the assump-

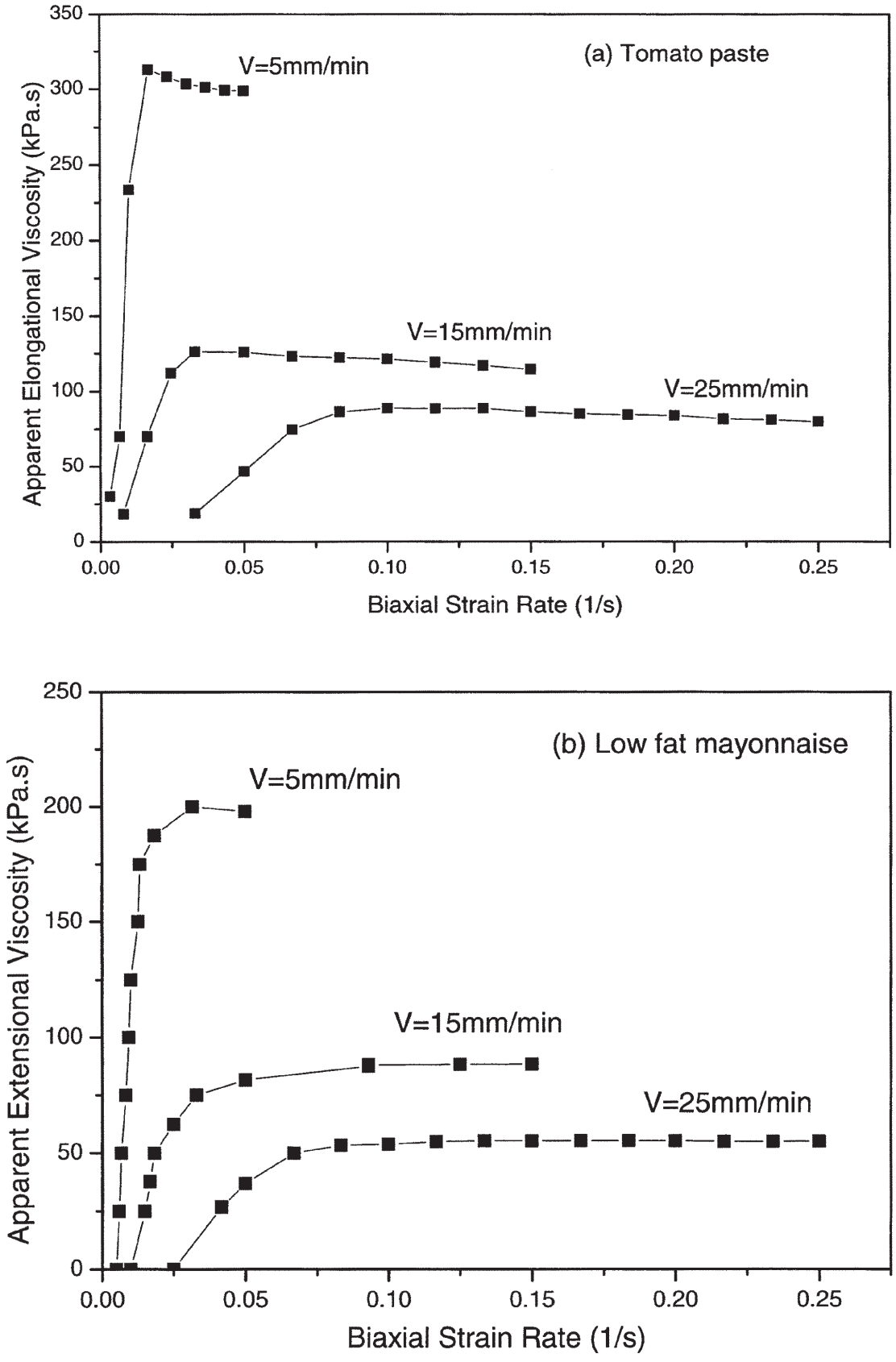


FIGURE 10. Experimental apparent elongational viscosity vs. biaxial strain rate relationships of tomato paste and mayonnaise at three displacement rates. Note the departure from the ideal behavior of a pseudoplastic fluid depicted in Figure 7.

tion that there is either an ideal friction or perfect lubrication. The concept of lubrication and/or friction during compression has been addressed many years ago (Forster 1955). In reality, as already mentioned, it is unlikely that full friction can be accomplished, especially if the tested food is self-lubricating. As to the opposite situation, even with well lubricated or Teflon-made plates, absolute frictionless conditions can never exist. Hence, the actual flow pattern is inevitably distorted, at least a certain degree, in all the test's varieties. (Had absolute frictionless conditions existed, it would be extremely difficult if not utterly impossible to mount the specimen!) Although there are mathematical tools to deal with a partial slip, how to quantify the effect of marginal friction on the flow ability of semiliquid foods is still an unsolved problem. Thus, even the best test's results should be treated as approximations only. Although a certain degree of uncertainty cannot be avoided, confidence in the results would significantly increase if they can be reproduced in tests performed under different conditions or with sensors of different geometries.

1. Transient Flow and End Effects

A fully developed squeezing flow regime can rarely be achieved instantaneously. Nevertheless, it is easy to identify the transient flow region and discard the corresponding part of the data (see Figure 8). It seems that the remaining part contains enough information to characterize the rheological behavior of the tested food if high compression ratios can be achieved. Because end effects are inevitable, it has been recommended that the tested specimen should have an aspect ratio of no less that 10 to 30 to reduce their relative weight (Leider and Bird, 1974b). Because by definition the aspect ratio increases as the specimen's height diminishes, this requirement also dictates that a high compression ratio must be reached for the results to be meaningful. Several reports on the squeezing flow of foods are based on smaller than the recommended aspect ratios and therefore how representative the data are is not always clear. For new food materials therefore it would be advisable to examine the effect of the aspect ratio on the calculated rheological parameters. Observations reported by Hoffner et al. (1997) and Suwonsichon and Peleg (1999 a,b,c) indicate that in mayonnaise, mustard, refried beans, and Ricotta cheese end effects are noticeable even at aspect ratios of above 50. The absolute magnitude of the end effects, however, is such that it does not interfere with the results interpretation, that is, the ranking of the foods compared by these authors was the same irrespective of the chosen plate diameter and hence the aspect ratio.

B. The Instrument's Stiffness and the Control of the Crosshead Speed and Location

When using plates with a diameter on the order of 100 mm, useful information is only obtained when the specimen reaches heights in the range of about 3 to 0.5 mm. This is a small displacement range that coincides with the region where the recorded force rapidly rises. Consequently, even a minor error in the plates positioning will result in a major error in the calculated rheological parameters, whatever they might be. The problem is potentially more serious in frictional squeezing flow viscometry where the force rise is approximately proportional to $(R/H(t))^3$ than in lubricated squeezing flow viscometry where the force is only approximately proportional to R/H(t). (Extending the range by further lowering the specimen's final height is not recommended because it can potentially damage the instrument.)

Every mechanical sensing device, be it a load cell or an LVDT, must deform somewhat in order to produce a signal. Because of the rigid construction of these devices, this deformation is commonly on the order of 0.01 to 0.001 mm and hence only a very minor source of error. The same applies to the electronic system's response time. In modern instruments the response time is sufficiently short, and hence has little effect on force measurements if the test is performed at a relatively small displacement rate. The instrument response time can become a significant factor if high speeds are used and the force increases very steeply. However, if the instrument itself has a significant compliance or if the speed and posi-

tioning controls are inaccurate and the actual crosshead motion is outside the specified range, the error can be quite large. When this happens even the general shape of the force-height relationship may appear distorted. Although some of these hardware problems can be corrected by various means, it is still highly recommended that squeezing flow viscometry be only performed with a testing machine of a robust construction and very accurate and tight controls.

C. Tilted Plates

The method as described in all its different configurations is based on the assumption that the plates are perfectly parallel. This is indeed an essential requirement. Recently, it has been shown that the effect of even a slight tilt progressively increases as the specimen height decreases, and with it the relative error (Hoffner et al., 2001). A sensor with almost perfectly aligned plates can easily be constructed and hence tilt need not be a problem. This is especially the case with plates whose diameter does not exceed 100 to 150 mm. However, it would still be prudent to check that the mounted plates are tightly connected to the instrument base and crosshead, and to verify that they are parallel with a level before any new experiments.

VI. OTHER POTENTIAL USES AND LIMITATIONS

The squeezing flow method's main advantage is that it enables testing foods practically intact. Hence, it also enables to test foods after controlled "abuse", before or after stirring, etc. Initial tests have shown that this can be done, and that it is possible to monitor how an "abused" or stirred specimen recuperates its consistency (Corradini et al., 2000a,b). As expected, it was found that a disturbed tomato paste, for example, does not fully restore its rheological properties even if left to rest for 3 hours. The sensory implication of a "disturbance" or "structural disruption" that may occur during handling is not fully known. However, again, the method, combined with sensory analysis, could reveal, at least in principle, what level of change in the foods rheological properties is also perceived, sensorily, as a textural change (Corradini et al., 2000a,b). The same procedure can be used to determine how a formulation, flavor, or any other factor affects the product consistency and its perception. Admittedly, research in this direction is only in its infancy. However, squeezing flow viscometry provides a useful tool for such investigations by allowing testing on the food in question practically intact initially, and then without added uncontrolled changes after the studied treatment.

The main limitation of the squeezing flow method, in all its varieties is that at least for the time being it is restricted to small displacement rates (see Table 2). Consequently, its results may not be useful to engineering design of operations that involve pumping, for example, where the rates are orders of magnitude higher. Also, the standard mathematical models, which had been primarily developed for polymers, may not be applicable to at least several important food types. Therefore, it would be a challenge to researchers in this field to develop new models that will be helpful not only in the interpretation of observed rheological behavior of semiliquid foods, but also in the prediction of their flow patterns in different geometries and rates. Even with these limitations, squeezing flow viscometry seems to be a practical and inexpensive solution to the two most serious problems of food viscometry today without any further development. The method can already be used for routine quality control in industry using available and relatively inexpensive instrumentation. As shown above, it can also help solving a variety of other problems for which shear-based viscometers are clearly inadequate despite their more sophisticated design.

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