CONSISTENCY OF DISPERSED FOOD SYSTEMS AND ITS EVALUATION BY SQUEEZING FLOW VISCOMETRY

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ABSTRACT

In testing semiliquid foods, conventional viscometers have two major problems: slip and disruption of the specimen's microstructure upon its insertion into the narrow gap of the sensor. These two problems are almost completely eliminated when lubricated squeezing flow viscometry is used; slip becomes a prerequisite for a proper test and, because of the large initial distance between the plates, the specimen can be tested practically intact. The penalty is that with the instrumentation commonly available in food research and quality assurance laboratories, the method can only be used at fairly small rates. In squeezing flow viscometry, the overall consistency of the tested material can be expressed in terms of the apparent stress at a given specimen height and its "degree of solidity" by the residual apparent stress at a given specimen height, measured after more than a minute at relaxation. The method is sensitive enough to determine differences between semiliquid foods, to record changes in their rheological properties as a result of shearing and to monitor their ability to recover their consistency when left to rest. The method also allows to follow the structural development of foams during their formation and collapse, because of drainage, when left undisturbed.

KEY WORDS

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INTRODUCTION

Liquid, semiliquid and solid foods form a continuous domain and the borderline between the groups has always been and will remain fuzzy. In contrast with liquid foods such as milk and juices, semiliquid food products like yogurt or mayonnaise usually do not, or only very slowly, flow under their own weight. At the same time, and in contrast with most "true solids," they are relatively easy to deform or spread and do not exhibit a brittle failure.

Concentrated or dense dispersed food systems are structured materials that present both solid and fluid rheological characteristics. Many are micro-heterogeneous products composed of two major phases, in which case the dispersed phase constitutes at least 20% of the system's mass (Metzner 1985). If both continuous and dispersed phases are liquids, we refer to the system as a concentrated emulsion. If solid particles are dispersed in a continuous liquid matrix, the system can be defined as a suspension. Although concentrated emulsions need not have solid constituents *per se*, the interconnection between the droplets and their packing makes the system solid-like. The same applies to suspensions, especially if the continuous phase can be considered as a weak gel.

From a rheological point of view, thick concentrated food systems are materials that have a substantial yield stress and a high apparent shear viscosity. These characteristics arise from the materials' structure, which also determines their time dependent rheological properties and the ubiquitous shear thinning that they exhibit. The existence of a yield stress in semiliquids has been a controversial issue in rheology, especially after the publication of Barnes and Walters' article (Barnes and Walters 1985; Barnes 1999). The controversy arises from reported experimental observations that liquids known to have a "yield stress" continuously deform under smaller stresses, albeit at such low rates, that the time to monitor the flow is on the order of weeks or months. Although the ensuing debate has fundamental implications, it should not concern us here. This is for the simple reason that most if not all foods are biologically, chemically and/or physically unstable. Hence, meaningful tests with such long duration are difficult and in some cases impossible to devise. This epistemological problem can be avoided or bypassed by entertaining the notion that certain foods respond to stress as if they had a yield stress *on the pertinent time scale*. Or in other words, such foods exhibit a degree of solidity that can be assessed in terms of an apparent yield stress that can be considered as real for all practical purposes.

The microstructure of many dispersed systems and its susceptibility to mechanical disruption has made it difficult to evaluate their consistency by conventional viscometric analyses. Moreover, to understand the relation between a bulk property, like consistency and the material's microstructural

characteristics, one needs to develop a method or methods to probe and assess the specimen's integrity in an undisturbed state.

The objectives of this short discussion article are to highlight the limitations of conventional rheological methods as a tool to assess the consistency of such systems and to demonstrate that squeezing flow viscometry can eliminate some of the problems that are associated with their mechanical testing.

CONSISTENCY EVALUATION OF SEMILIQUID FOODS

Empirical Methods. The Bostwick consistometer, back extrusion cells, penetrometers, etc., have been widely used to characterize dense dispersed food products, particularly tomato products such as pastes and purees, fruit juice concentrates and butter or margarine. Although these tests offer a simple and convenient way to compare samples, their results cannot be expressed in terms of universal units, primarily owing to the instrument's arbitrary geometry (Steffe 1996). Hence, the meaning of differences between samples is hard to assess, except on the basis of actual experience with the particular product.

Shear Viscometry. Conventional coaxial (including cone and plate parallel plates) rheometers have been extensively used in an attempt to characterize the consistency of semiliquid food products and, to a lesser extent, capillary viscometers. However, there are several sources of error when such instruments are used regardless of their sensor's geometry.

Solid particles within the dense dispersed system tend to migrate to regions of low shear stress upon application of shear (Leighton and Acrivos 1987; Abbot *et al.* 1991; Mondy *et al.* 1994; Phan-Thien *et al.* 1995) resulting in the formation of a film devoid of particles at the rheometer's sensor walls. Phase separation can also occur if there are noticeable differences in density among the system's components (Van Wazer 1963; Goshawk *et al.* 1998). Owing to the "slip" which results, the measurements may reflect the consistency of the separated layer rather than that of the bulk material which is not sheared to the extent required for a valid rheological analysis. Slip has been observed in all coaxial instruments, irrespective of the sensor type and geometry, and in capillary rheometers as well (Yoshimura and Prud'homme 1988a; Plucinski *et al.* 1998; Pal 2000). Contradictory results have been reported as to the effect of slip on the results of oscillatory tests (Yoshimura and Prud'homme 1988b; Ma and Barbosa-Canovas 1995; Goshawk *et al.* 1998) but there is little doubt that it can affect measurements of this kind too.

It is difficult to place a semiliquid specimen in the narrow gap of a conventional viscometer's sensor without disrupting its structure to at least some extent because most concentrated food emulsions and suspensions have

a relatively weak structure. The actual amount of structural damage depends on the specimen's shear history during its loading and handling, which can vary dramatically among instruments and operators. Consequently, rheological measurements with conventional shear rheometers are frequently affected by an uncontrolled and unknown degree of structural alteration that is impossible to quantify.

Consistency loss, as a result of shearing, was studied by Tiu and Boger (1974) and Halmos and Tiu (1981). They reported how the rheological properties of mayonnaise and meat extracts are influenced by the sample's shear history and proposed mathematical models to quantify the effects. It is a commonplace observation that the apparent viscosity of semiliquid foods that exhibit shear thinning (pseudoplasticity) is also time dependent. The shearing-history effect can be determined directly in terms of the apparent viscosity loss. It can also be extracted from the area of the hysteresis when the flow curve is recorded at increasing and then decreasing rate (The reader will notice that an enclosed area on shear stress – shear rate coordinates has the units of power per unit volume). All such measurements do not account for the initial structural disruption that occurs when the specimen was inserted into the viscometer's sensor.

A common practice in viscosity determinations of semiliquid materials is to let the sample rest in the sensor prior to starting the measurement itself to allow the specimen to recover its original consistency. If the structural disruption is reversible, the consistency might be fully recovered within a reasonable time, i.e., on the order of minutes to hours. If, however, the damage is irreversible, as in the notable case of the casein gel in yogurt, the loss of consistency would be permanent. Another possibility is that the consistency can only be partially recovered, in which case the recovery's extent would depend on the time that the specimen has been allowed to rest. In a semiliquid food tested for the first time, one should expect only partial recovery to an unknown degree (Corradini *et al.* 2000a). Even if the specimen could fully recover its original consistency, it would be still difficult to determine the proper rest time without extensive experimentation. The required rest time might be on the order of several hours to a whole day, in which case other alterations might occur in the specimen as a result of dehydration, syneresis or microbial activity.

Finally, the presence of solid particulates of a size on the order of the gap found between the sensors' moving part and walls can also interfere with the flow pattern. If the particles are large enough, they might clog the instrument's sensor thereby rendering the test irrelevant.

Lubricated Squeezing Flow Viscometry. Lubricated squeezing flow viscometry (Fig. 1), originally introduced to foods by the group of Bagley at
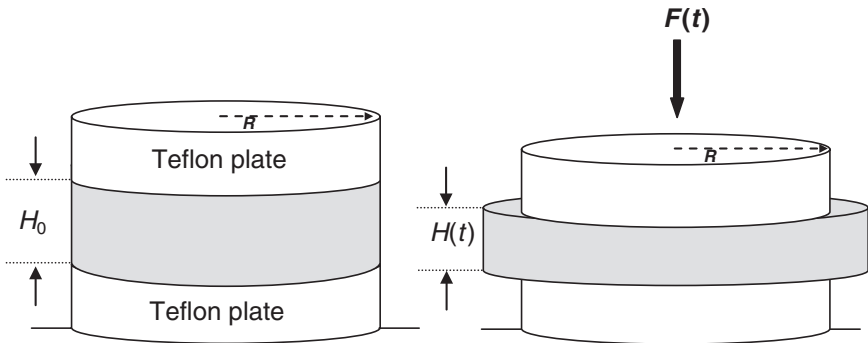
FIG. 1. SCHEMATIC VIEW OF A SQUEEZING FLOW ARRAY

the USDA laboratories in Peoria (Casiraghi *et al.* 1985), offers a way to overcome most of the above problems. It enables the determination of the rheological properties of materials that violate the no-slip boundary condition, which is essential for the traditional viscometric methods. If the specimen's self-lubrication is insufficient, friction can be almost totally eliminated by covering the plates with a lubricating oil of low viscosity or by using Teflon sensors (Instron Corp., Norwood, MA). Slip in this testing method is no longer a problem that needs to be overcome but a prerequisite for a proper test.

The method offers a practical way to avoid the extensive structural disruption that would have occurred had the specimen been forced into a narrow space because loading of the sample in the squeezing flow viscometry is performed when the plates are widely separated. The damage can be almost completely eliminated and the specimen can be tested as practically intact by carefully placing the material on the lower plate with a wide spatula or spoon (Suwonsichon and Peleg 1999a; Corradini *et al.* 2000b).

Finally, the presence of suspended particles, unless of a size on the order of the final separation between the plates, hardly affects the test performance (Suwonsichon and Peleg 1999a).

Procedure and Data Analysis

Squeezing flow viscometry, be it lubricated or frictional, has several variants with respect to geometry, namely, constant volume and changing area or constant area and changing volume (Fig. 2). The test itself can be performed at a controlled displacement rate, measuring the force-time relationship, or under a constant load (creep) where the height-time relationship is monitored, (Campanella and Peleg 2002) – see Fig. 2. Another option is to perform dynamic squeezing flow test, i.e., to subject the specimen to small axial strain

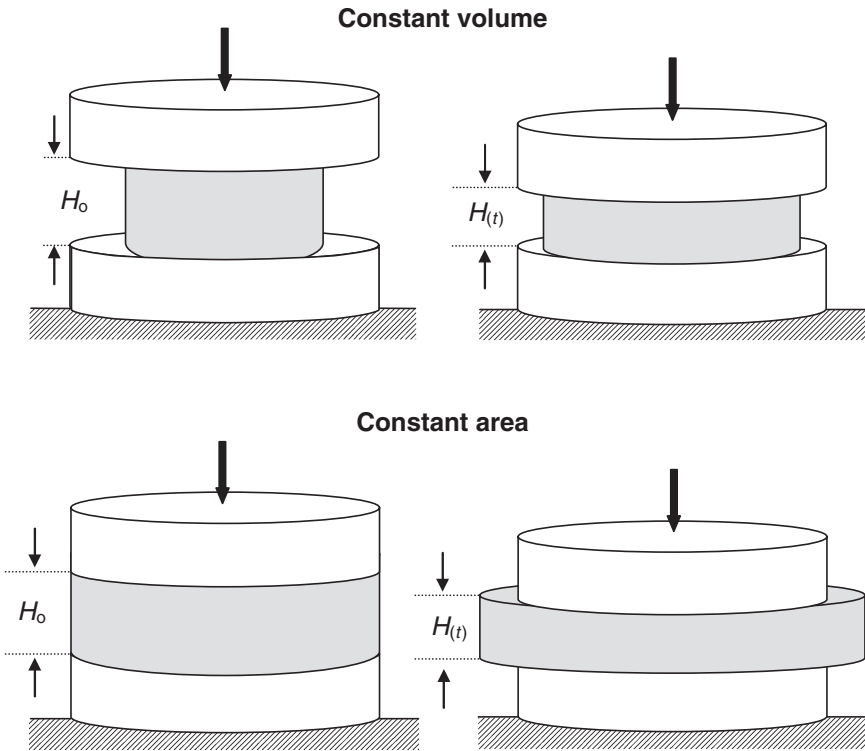**Constant volume**

Constant area

FIG. 2. THE GEOMETRY OF A LUBRICATED SQUEEZING FLOW TEST PERFORMED AT
CONSTANT AREA AND DISPLACEMENT RATE
Notice that in lubrication flow (in contrast with frictional flow) the front of the expelled fluid is flat.

oscillations (e.g., Phan-Thien *et al.* 1996), measuring the in phase and out of phase stresses. The interpretation of the results of such tests is straightforward when the specimen exhibits linear viscoelasticity and hence the very small strains. The usefulness of dynamic methods in the consistency of assessment of *viscoplastic* foods that undergo structural changes at large strains is yet to be demonstrated. Their application would require expensive instrumentation that can only be found in a few industrial and academic research centers but not in ordinary quality control laboratories, even if they could be used to characterize the intact structure of semiliquid foods.

    Owing to the difficulties to form a regular and stable specimen of semi-liquid foods, the constant area and changing volume array is usually preferred (Fig. 2). This is in contrast with many synthetic polymers where preparing a specimen in the form of a disk is easy. In constant area – changing volume

configuration, because the specimen's momentary height is measured from the sensor's bottom – any irregularities in the specimen's initial height and even the initial height itself primarily influence the transient flow regime (see Fig. 3). Data generated in this regime are discarded and the fully developed squeezing flow regime, which is only accomplished at high compression ratios (specimen height ≤3 mm in sensors of 60–140 mm in diameter), is hardly affected.

The sensor itself can be mounted on almost any rigid universal testing machine. These are widely used in food research and quality control laboratories, and hence, the constant area – the constant displacement rate version of the test is the most convenient and commonly used. In the majority of non-Newtonian foods, it is easier to produce conditions that are closer to ideal, lubricated squeezing than to guarantee the type of contact with the plates that will produce "slipless" frictional flow. In some materials, notably peanut butter, self-lubrication is difficult if not impossible to avoid. The test itself is easy to perform and the raw data, in the form of force-time files, can be converted into force-height or log force versus log height relationships using standard office software. Typical plots of these relationships for a semiliquid food system are presented in Fig. 3. It shows that plotting the force-height relationships on logarithmic coordinates enables one to identify the displacement levels where a fully developed squeezing flow is achieved. The flatness of the squeezed specimen's front provides visual evidence that the flow is lubricated. But there are also other ways to establish that friction has been almost completely eliminated – see below.

Most dense dispersed food systems behave as pseudoplastic materials, at least superficially. The equation that governs the flow of ideal pseudoplastic fluids in a constant area-changing volume configuration has been developed by Campanella (Campanella 1987; Hoffner *et al.* 2001):

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

where $F(t)$ and $H(t)$ correspond to the momentary force and height, respectively, R, the plates' radius, K, the consistency coefficient, n, the flow index and V, the displacement rate.

The apparent flow index, n, can be inferred from the absolute magnitude of the slope of the log $F(t)$ versus log $H(t)$ relationship at a constant displacement rate (Eq. 1). When that value of the determined n is smaller than one, it is almost certain that the flow is predominantly frictionless. If the absolute slope is close to three (the maximum theoretical value, i.e., of a Newtonian fluid), the flow is clearly frictional (Campanella and Peleg 2002). Even in such case the possibility of partial slip cannot be ruled out and it would be difficult
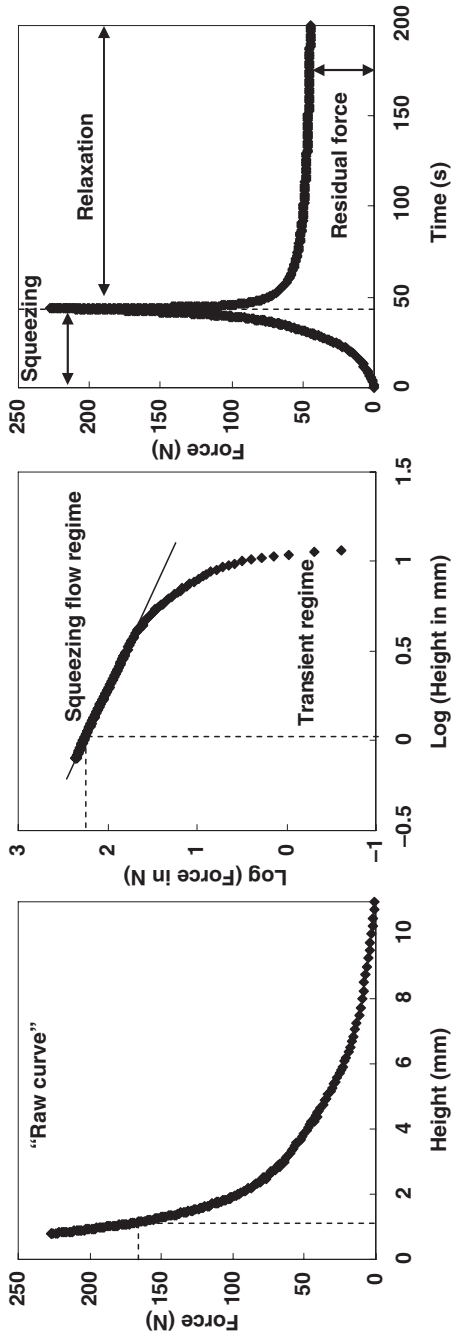
FIG. 3. TYPICAL FORCE-HEIGHT, LOG FORCE-LOG HEIGHT AND FORCE-TIME RELATIONSHIPS OF A "THICK" SEMILIQUID FOOD PRODUCT

Notice the appreciable residual force after relaxation and that the squeezing flow regime can be easily identified.

to decide whether the magnitude of the observed power, n, is a correct measure of the flow index. In lubricated squeezing flow, in contrast, once the frictionless character of the flow has been established, the nature of the liquid can be assessed from the slope of the log $F(t)$ versus log $H(t)$ relationship. If $n = 1$, the liquid is Newtonian, and if $n < 1.0$, it is pseudoplastic. And, once n has been determined, the apparent K can be calculated directly with Eq. (1).

In principle, a lubricated squeezing flow test also allows one to calculate the fluid's elongational or biaxial viscosity (Chatraie *et al.* 1981), i.e.:

$$\mu_b = \frac{\sigma_b}{\dot{\varepsilon}_b} = \frac{2F(t)H(t)}{\pi R^2 V} \tag{2}$$

where μ_b, is the elongational viscosity, σ_b, the biaxial stress and $\dot{\varepsilon}_b$ the biaxial strain rate.

Theoretically, the apparent μ_b of a Newtonian liquid is three times its shear viscosity. In a pseudoplastic fluid, μ_b is still a material property, albeit a rate dependent one. When calculated in a variety of semiliquid food products, it was found not to be a unique function of the strain rate as the equation for pseudoplastic fluids entails. This could be primarily attributed to these products' considerable *plasticity*, a result of structural changes that might occur during the specimen's deformation. The rate effect could be described in terms of an empiric model, based on the assumption that only the relaxing component of the stress is rate dependent, while the residual unrelaxed portion is not (Suwonsichon and Peleg 1999b; Corradini *et al.* 2000b).

The apparent compressive stress at any chosen specimen height H, $\sigma_{App@heigtH}$, within the squeezing flow regime, can be used as a measure of the product's overall consistency regardless of its more subtle rheological characteristics.

It is calculated as:

$$\sigma_{App@heightH} = \frac{F_{@heightH}}{\pi R^2} \tag{3}$$

The residual stress after relaxation (see Fig. 3) can serve as an empirical measure of the sample's degree of solidity. The residual unrelaxed stress is most probably related to the sample's apparent yield stress and calculated as:

$$\sigma_{App@timet} = \frac{F_{@timet}}{\pi R^2} \tag{4}$$

where $F_{@heightH}$ and $F_{@timet}$ are the forces at the chosen height and after a given time at relaxation, respectively.

Although lubricated squeezing flow viscometry has an obvious advantage over conventional rheometers with respect to slip and premature disruption of the specimen's microstructure, it has its own serious limitation. With the current generation of testing machines, as already mentioned, the specimen can only be deformed at relatively low biaxial rates. Consequently, the test results cannot be used to calculate flow properties that are pertinent to pumping or other engineering operations, where high shear rates are involved.

A fully developed squeezing flow regime cannot be achieved instantaneously, as already stated. However, the region of transient flow regime can easily be identified on the log force versus log height plot (Fig. 3) and the corresponding data discarded. Yet, in squeezing flow viscometry, high compression ratios are essential for meaningful test results. Any small error in the recorded height will produce a very large error in the determined force because in this region the force ascent is very steep. Consequently, only testing machines of robust construction and having a very accurate control of the crosshead movement should be used if artifacts are to be avoided.

Another requirement for a valid squeezing flow test is that the plates are as close as possible to being perfectly parallel. Even a slight tilt can cause a significant error, which would increase progressively with the compression ratio (Hoffner *et al.* 2001).

CHARACTERIZATION OF DENSE DISPERSED FOOD SYSTEMS

Concentrated Emulsions: Mayonnaise and Salad Dressings. Mayonnaise normally contains at least 65% vegetable oil dispersed in the form of small fluid droplets. "Spoonable" salad dressing, with a mayonnaise-like texture, contains 35–50% oil and with starch as a thickener. "Pourable" salad dressing may contain even less oil and tends to undergo phase separation (Potter 1986).

The consistency of semiliquid dressings is mainly determined by their dispersed phase volume fraction; the higher the oil content, the "stronger" the consistency. However, the droplets' packing and the continuous phase's viscosity also play an important role (Holcomb *et al.* 1990) and hence their manipulation is used to control the rheological properties of such products.

The dispersed phase in emulsions, in contrast with suspensions, consists of fluid particles. Consequently, the droplets' internal circulation (wherever possible) and deformability can also affect the system's consistency in a different manner. In concentrated emulsions, the droplets are close to each other and may interact strongly while in a very dilute emulsion, the droplets are relatively far apart and their interactions relatively weak. Extensive aggre-

gation of the closely packed droplets in dense dispersed food emulsions generates a gel-like network (Dickinson 1989), which not only improves their stability but also confers textural properties that in most cases are considered desirable.

The merits and drawbacks of mayonnaise consistency assessment with conventional shear rheometers have been extensively discussed. Plucinski *et al.* (1998) observed slip in mayonnaise tested in both coaxial and tube viscometers. Slip in rotational viscometers may render them ineffective in the determination of the mayonnaise's consistency and according to Plucinski *et al.* (1998) "tube viscometers can yield meaningful results only at high shear rates for this reason." Extensional viscometry has therefore been proposed as an alternative. Using spectroscopic techniques, Goshawk *et al.* (1998) confirmed that there were structural and compositional differences in the material at the wall of the viscometer compared to that at the center in a sheared sample exhibiting slip. Bower *et al.* (1999) tried to relate the observed rheological properties of mayonnaise during shearing with microstructural alterations that occur in the specimen. They suggested that slip causes changes in the droplet size distribution and induces local microscopic flows, and these were probably responsible for the discrepancy between the observed and expected rheological properties of the mayonnaise.

Additional sources of error in the rheological characterization of concentrated emulsions like mayonnaise are edge fracture, progressive expulsion of the sample from the sensors and irreversible structural breakdown (Campanella and Peleg 1987; Barbosa *et al.* 1996). A transient flow regime characterized by a stress overshoot has been also observed in mayonnaise tested with conventional rotational viscometers (Kokini and Dickie 1981; Campanella and Peleg 1987). Whether the slip occurs only in steady shear testing but not in oscillatory (dynamic) tests is still a debatable issue (Ma and Barbosa-Canovas 1995; Goshawk *et al.* 1998).

Squeezing Flow. The effect of the mechanical history on an emulsion's consistency and degree of solidity can be monitored by testing them in squeezing flow before and after compression and/or shearing. The results can then be expressed in terms of the apparent stress at a given height (in our case, 1-mm height, see below) and after a given time (2 min in our case). Comparison of such measures in mayonnaise and salad dressings is shown in Table 1.

The mayonnaise's stronger consistency as compared with that of the salad dressing is clearly evident in the overall magnitude of the stresses before and after stirring (in a domestic blender for 1 min). The disruption of the sample by its compression between two Teflon plates had produced only minor loss of consistency. In both the mayonnaise and salad dressing, letting the stirred samples rest, even for an hour, resulted in only partial recovery of their

TABLE 1.
RHEOLOGICAL PARAMETERS OF SEVERAL PRODUCTS DETERMINED WHEN THEY WERE PRACTICALLY UNDISTURBED AND AFTER CONTROLLED MECHANICAL DAMAGE AND RECOVERY

Product	State	Apparent stress @1 mm (kPa)	% Consistency loss/Recovery	Apparent stress after 2 min (kPa)	References
Mayonnaise ~60% fat	Practically intact	10.0–9.0	–	3.0–2.5	Corradini et al. (2000a)
	Compressed	9.5–9.0	3.0–4.0	3.0–2.5	
	Stirred	8.0–7.0	17.0–20.0	2.5–2.0	
	After 1-h rest	9.0–8.0	40.0–45.0	3.0–2.5	
Salad dressing ~40% fat	Practically intact	6.0–4.0	–	1.0–0.0	Chanasattru et al. (2002)
	Stirred	4.0–2.0	20.0–30.0	0.5–0.0	
	After 1-h rest	4.0–3.0	5.0–10.0	0.5–0.0	
Mustard	Practically intact	4.5–4.0	–	1.0–0.5	Corradini et al. (2000a)
	Compressed	4.0–3.0	10.0–15.0	0.0–0.5	
	Stirred	2.0–3.0	30.0–40.0	0.0–0.5	
	After 1-h rest	2.0–3.0	0.0	0.0–0.5	
Mustard w/seeds	Practically intact	4.0–3.0	–	2.0–1.0	Suwonsichon and Peleg (1999b)
Tomato paste (30°Bx)	Practically intact	17.0–16.0	–	6.0–5.0	Corradini et al. (2000b)
	Compressed	16.0–15.0	5.0–8.0	5.0–4.0	
	Stirred	14.0–13.0	15.0–20.0	3.0–4.0	
	After 1-h rest	14.0–13.0	0.0–5.0	4.0–5.0	
Tomato paste (27°Bx)	Practically intact	11.0–10.0	–	3.0–5.0	Corradini et al. (2000b)
Tomato paste (22°Bx)	Practically intact	8.0–5.0	–	6.0–3.0	Lorenzo et al. 1997
Tomato puree*	Practically intact	2.0–3.0	–	0.5–0.6	Lorenzo et al. (1997)
Ketchup (32°Bx)*	Practically intact	1.5–1.3	–	1.0–0.9	Lorenzo et al. (1997)
Bean paste	Practically intact	15.0–10.0†	–	6.0–3.0	Suwonsichon and Peleg (1999c)
Chick pea paste	Practically intact	30.0–23.0	–	7.5–6.0	Kampf and Peleg (2002)
Traditional	Stirred	13.0–8.0	70.0–45.0	3.5–1.5	
Chick pea paste	Practically intact	10.0–7.0	–	1.0–0.2	
Commercial	Stirred	4.5–4.0	60.0–35.0	0.4–0.35	
Peanut butter	Practically Intact	30.0–25.0	–	16.0–15.0	Unpublished data

* Determined using a different geometry (imperfect squeezing flow, in which one of the plates is replaced by a shallow container) owing to the fluidity of products tested.
† Apparent stress at 1.5 mm.

rheological properties. Apparently, the high shear during the stirring was responsible for partial coalescence of the oil droplets, which was an irreversible process, while the droplets' deformation and rearrangement (flocculation – defloculation) according to Dickinson (1992), is a reversible process. Moreover, there was a noticeable difference in the degree of recovery between the mayonnaise and salad dressing. In mayonnaise, spontaneous structural reformation can occur, but not so in the salad dressing and at least not on the reported time scale. This observation is supported by the recovery of the stress overshoot in tested mayonnaise after 24 h (Campanella and Peleg 1987).

Concentrated Suspensions

Mustard. Most mustards consist of finely ground mustard seeds suspended in an aqueous medium. Some may also contain whole or coarsely ground seeds. The total amount of suspended solids in these systems is usually 15–20% by weight, but because of the swelling of the solid particles, their volume fraction is considerably higher (Aguilar *et al.* 1991a). The suspended hydrated particles form a network structure in which the liquid phase is trapped. Agitation may produce a more compact structure from which the liquid is expelled causing syneresis. The phenomenon can also occur spontaneously during storage resulting in visible separation of liquid floating on the mustard (Aguilar *et al.* 1991b). The weakness of the mustard's microstructure, its instability and tendency to expel entrapped liquid can become a problem in its consistency evaluation by viscometeric methods especially when based on shear.

Some rheological characteristics of mustard obtained by lubricated squeezing flow viscometry are shown in Fig. 4 and Table 1 which demonstrates that the mustard's weak structure can be disrupted to a considerable extent even by the small stresses that are produced during compression between Teflon plates. Although the reported consistency of samples with and without whole seeds was similar, the apparent stress after the relaxation period was much higher in samples that contained the seeds. Whether this was because of the seeds' presence or to a network strengthened by other ingredients, is yet to be clarified. The table also shows that once disrupted by shear, the mustard cannot regain its original rheological properties even after 1 h of rest.

Tomato Products. Tomato juice, sauce, puree and paste as well as ketchup, consist of disintegrated pericarp cells suspended in a clear serum. The total soluble solids concentration in tomato juice is 4 to 5%, in puree and sauces 8% to 24% and in pastes, at least 24% (Barrett *et al.* 1998). The solubles comprise of sugars, aminoacids, organic acids, salts and pectic substances. Although the total soluble solids content affects the textural characteristics of
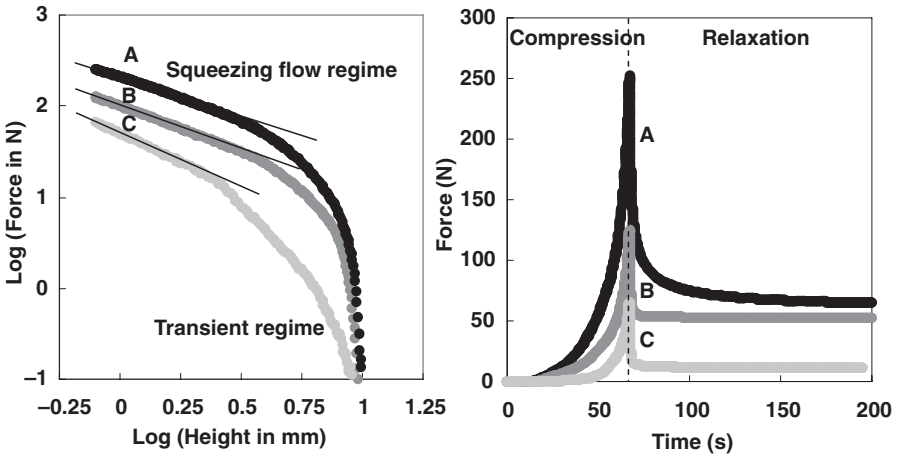
FIG. 4. COMPARISON OF THE TYPICAL LOG FORCE-LOG HEIGHT AND FORCE
RELAXATION CURVES OF TOMATO PASTE (A), MAYONNAISE (B) AND MUSTARD (C)

tomato products, their consistency is mainly determined by the pectin's integrity and the insoluble solids concentration. Intact or crushed cells and long-chain biopolymer, such as cellulose, hemicellulose and water insoluble pectin, constitute this water insoluble portion (Hayes *et al.* 1998).

The suspended solids' particle size distribution and shape also affect the consistency of tomato products. A reduction in particle size is expected to result in a product with a stronger consistency partly because of an increase in the strength of the particle–particle interactions and possibly because of the more effective release of the pectin. According to some literature sources, large elongated particles, which have the ability to form aggregates and networks, are also a crucial element in maintaining the consistency of tomato products (Rao and Cooley 1992; Hayes *et al.* 1998; Rao 1999; Sanchez *et al.* 2002).

Serum separation can affect the rheological measurements of all types of tomato products. Premature shearing can disrupt both the pectin's gel structure and the suspended particle aggregates, thus altering the whole network integrity. The results of consistency determination of tomato products obtained by lubricated squeezing flow are reported in Table 1. The higher content of soluble solids correlates with a higher consistency, as could be expected. Figure 5 depicts the effect of different manners of mechanical disruption on the squeezing flow curves of tomato paste. Compression between Teflon plates, which hardly produces any shear, had little effect on the apparent consistency of the paste. Stirring, which produces intensive shearing, caused a much more extensive loss of consistency. Dif-
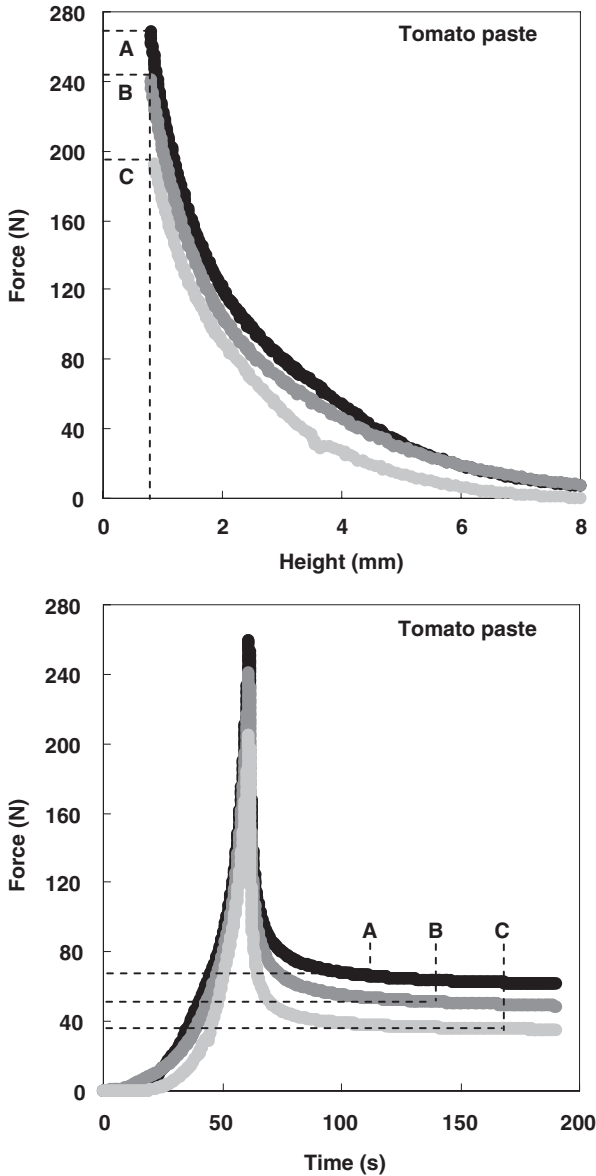
FIG. 5. EFFECT OF MECHANICAL DISRUPTION ON THE SQUEEZING FLOW CURVES OF
TOMATO PASTE
(A) The paste measured practically intact, (B) after compression between parallel teflon and (C)
after stirring (shear).
Notice that the differences are clearly of a measurable magnitude.

ferent levels of stirring (see Fig. 5) cause different degrees of consistency loss, which can be easily detected and measured by the method (Corradini *et al.* 2000b).

Total restoration of the original consistency has never been achieved in any of the tomato products tested (Corradini *et al.* 2000b). Even after an hour of rest, the recovery was barely measurable. More substantial recovery could be observed after 3 h of rest, but the practicality of leaving a sample for so long in routine industrial testing is highly questionable.

Bean Pastes, Chickpea Pastes and Peanut Butter. Chickpea and bean pastes (Mexican style) have a significant amount of suspended, noncolloidal solids (30%), and a lipid phase (~10%) dispersed in an aqueous matrix. Little is known about the microstructure of these systems. Kampf and Peleg (2002) analyzed chickpea pastes obtained by different preparation methods and of different composition. As in the case of other semiliquid foods of plant origin, the exact effect of the suspended solids' particle size distribution on the rheological behavior of these suspensions has not yet been fully revealed (Barrett *et al.* 1998). The samples with the higher concentration of suspended particles did not necessarily exhibit a stronger consistency. Apparently, the emulsion's matrix rather than the suspended particles had the dominant effect on the overall product's consistency, as well as on their degree of "solidity."

Peanut butter is a suspension of small noncolloidal peanut particles in peanut oil (Citerne *et al.* 2001). Proteins as well as cell wall fragments are well dispersed in a continuous oil matrix that may contain other vegetable oils (e.g., palm oil) and stabilizers (Aryana *et al.* 2000). The solids' volume fraction has been estimated to be about 0.6, close enough to the theoretical maximum packing density. Hence, particle–particle interactions can have a significant effect on the products' rheological properties even without the other ingredients' contribution.

Owing to the lipid matrix, peanut butter consistency is difficult to characterize by traditional methods. Without a stabilizer, oil separation can be even visually observed. Slip is therefore a common problem in rheological evaluation of peanut butter by any method that involves shearing, regardless of the sensor's geometry and of whether the test is based on steady, monotonically changing or oscillatory shear stress or strain (Citerne *et al.* 2001). The effect of self-lubrication can be reduced by using serrated sensors or with the sensors having their surfaces roughened by other means. Although such sensors reduce slippage, it is unclear whether they can eliminate it completely, at least in peanut butter testing.

Many Mexican-style bean pastes have particles on the order of 2–3 mm in size and these can clog the sensor of a conventional bob-and-cap viscometer.

Such products however, can be easily tested in a squeezing configuration. The rheological parameters determined in a lubricated squeezing flow are presented in Table 1.

Among the above-mentioned products, peanut butter and the traditional chickpea paste had an exceptionally strong consistency as compared with other semiliquid foods. This is expressed in the apparent stress at 1-mm height on the order of 22–30 kPa, about twice as that of the other products. They also maintained a significant residual stress after relaxation indicating a relatively high degree of "solidity." Even mild stirring (with a spatula for 30 s) was enough to disrupt the microstructure of the chickpea pastes to a significant extent. An accompanying substantial drop in the apparent residual stress after 2 min of relaxation time was also observed in this case, consistent with the notion that the chick pea paste's network is mechanically unstable.

Foams

A liquid foam is a dispersed system where air or other gas bubbles constitute the discontinuous phase. A foam's rheological characteristics can be inferred from the bubble's deformability and its relation to the mechanical properties of the liquid film that holds them together (Wearie and Hutzler 1999). If the bubbles are very small relative to the size of the tested specimen, the foam can be considered as a continuum despite its discernible structure. The two approaches are not mutually exclusive and the choice of one *vis-á-vis* the other largely depends on the application. When viewed as a continuum, a foam can be tested by the same methods that are used for other dispersed systems. Attention should be paid to the possibility that the bubble size distribution, unlike that of dispersed solid particles, can change appreciably on the time scale of the specimen's preparation and sometimes even on that of the test itself.

Examples of typical force-time curves of egg albumen foams fortified with two gums – propylene glycol alginate and xanthan – are shown in Fig. 6. Figure 6 and Table 2 show how the foam's rheological properties change during the foam's formation and when left to rest. In this case, the changes were primarily a consequence of drainage which accompanied a change in the bubble's size distribution as demonstrated in Fig. 7 (Kampf *et al.* 2003). The log $F(t)$ versus log $H(t)$ plots of the fully formed foams (after at least 2 min of "beating") had a slope on the order of −0.7 to −0.8, which indicated that the flow was indeed "lubricated". The method, as the above indicates, has been sensitive enough to monitor the consistency development during the foam's formation, how it was affected by the gums addition and how it spontaneously diminished as a result of the bubble's collapse and the liquid film's drainage.
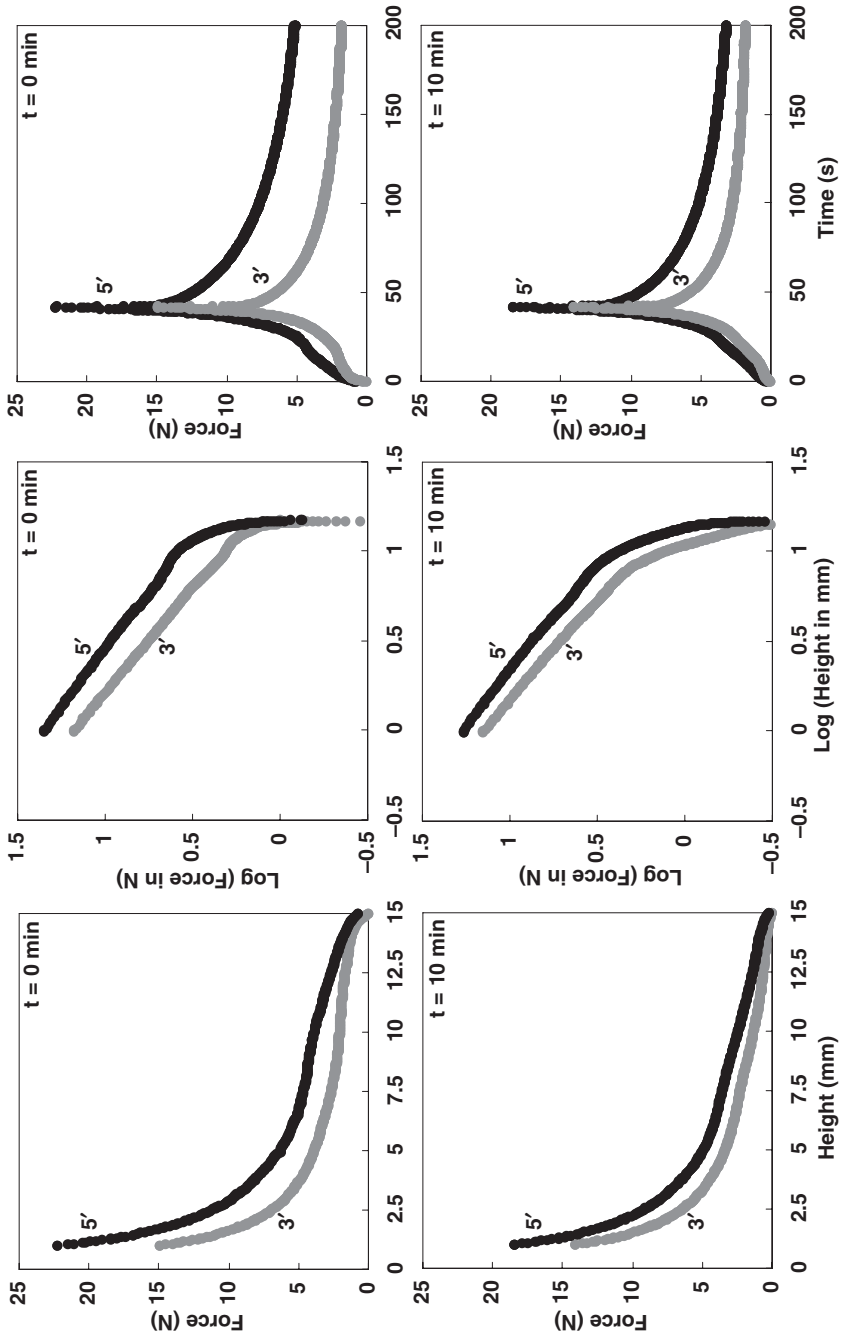
FIG. 6. LUBRICATED SQUEEZING FLOW CURVES OF EGG ALBUMEN FOAMS "BEATEN" FOR 3 AND 5 MIN IMMEDIATELY AFTER FORMATION (TOP) AND AFTER 10 MIN OF REST

Data are from Kampf *et al.* (2003).

Notice that both the foam's development and stability can be monitored by squeezing flow viscometry.

TABLE 2.
RHEOLOGICAL PARAMETERS OF ALBUMEN AND ALBUMEN-GUM FOAMS AT DIFFERENT MIXING TIMES[1]

Gum		Mixing time (min)	Slope	Apparent stress after 1 mm (kPa)	Apparent stress after 2 min (kPa)
None	Immediately after formation	3	0.81	2.10	0.36
		5	0.74	2.80	0.97
	After 10-min rest	3	0.81	1.90	0.29
		5	0.78	2.50	0.58
Propylene glycol alginate	Immediately after formation	3	0.73	2.50	0.52
		5	0.70	4.60	2.10
	After 10-min rest	3	0.74	2.40	0.50
		5	0.72	3.40	1.40
Xanthan gum	Immediately after formation	3	0.70	2.60	0.70
		5	0.69	4.20	2.00
	After 10-min rest	3	0.75	2.40	0.65
		5	0.73	3.00	1.10

[1] The data are from Kampf *et al.* (2003).

Bubble size distribution immediately after formation
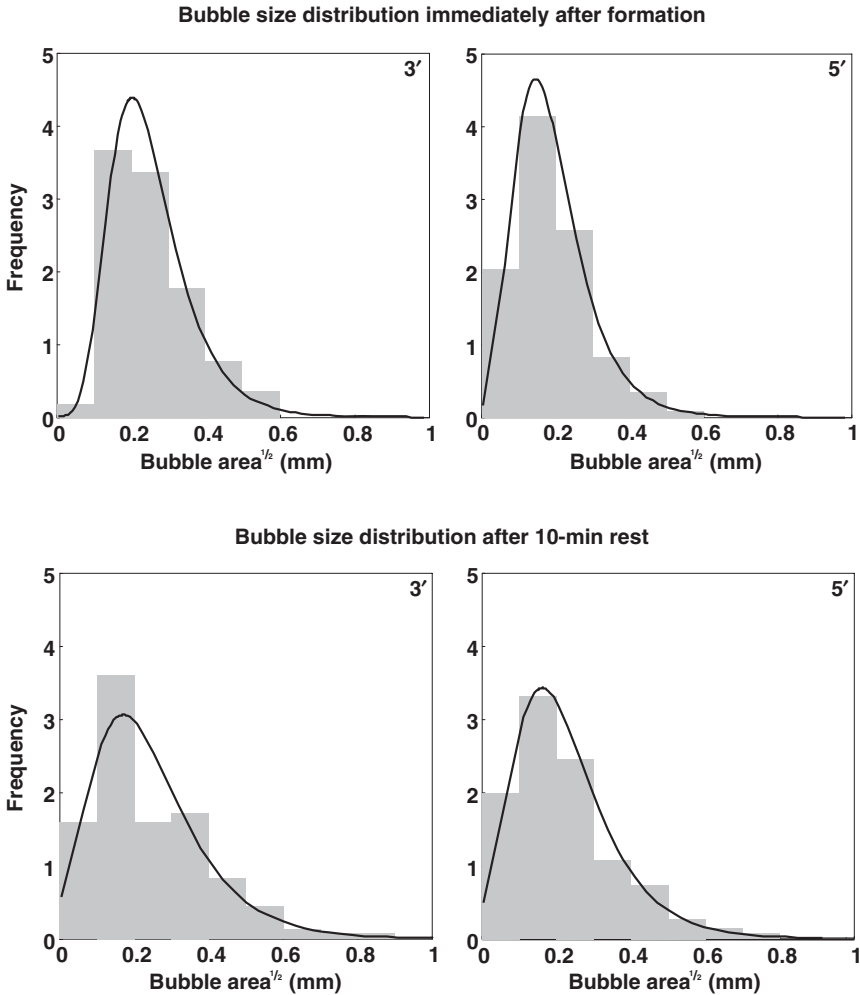

Bubble size distribution after 10-min rest

FIG. 7. CHANGES IN THE BUBBLE SIZE DISTRIBUTION OF THE EGG ALBUMIN FOAMS,
PRODUCED BY "BEATING" FOR 3 AND 5 MIN
Notice that "stability" and "consistency" can be associated with a narrow and small bubble size
distribution (compare Fig. 6).
The data are from Kampf *et al.* (2003).

Table 2 shows that the reproducibility of the measurements left little doubt that
the observed changes in the rheological characteristics were the result of the
structural alterations, which were observed simultaneously through micro-
scopic examination. They could still be monitored by the ordinary squeezing

flow test procedure despite the relatively fast rate of the changes in the foam's structure. It is doubtful that similar results could be obtained with traditional viscometers. This is because the mere insertion of an unstable foam into the narrow gap of the sensor, or merely its compression upon the specimen loading would most probably cause considerable disruption of the foam's delicate microstructure.

CONCLUSION

All the described semiliquid foods have an internal structure that can be easily disrupted by applied shear. Therefore, their consistency must be determined by methods that minimize structural damage prior to the test's commencement. Lubricated squeezing flow viscometry offers as a practical alternative to conventional rheometry based on shear by allowing testing the specimen almost intact. Disruption while loading the specimens onto the sensors has only a minor effect on the results. This has been evident from the reproducibility of the stresses measurements on the order of about 10% or less when expressed as a coefficient of variation, which is only slightly higher than that observed in oils testing (Suwonsichon and Peleg 1999b). The method has proven useful in quantifying the overall consistency of a variety of semiliquid foods and foams with very different composition and structure on the same scale. It also allows to separately determine such products' consistency, i.e., its overall resistance to deformation, which is expressed by the stress at a given specimen height, and the product's degree of "solidity," which is manifested in the apparent residual stress after relaxation. Although not reported in this article, the method has also been sensitive enough to detect textural differences between semiliquid foods of different brands and even batches of a food of the same brand that were produced on different dates. It enables monitoring and quantifying the effects of mechanical abuse on the products' consistency. Compression between Teflon-coated plates alone has only a minor disruptive effect in comparison to even mild shear. Thus, avoiding shear during the sample handling and loading should be of primary concern to those evaluating the texture of semiliquid foods by traditional methods.

Rheological parameters obtained by the method can be used to identify the mechanical properties that are actually perceived sensorily because in squeezing flow viscometry, the specimen is tested practically intact. This can be done by testing and tasting samples of the same food that only differ in their texture, but not in other sensory property (Chanasattru *et al.* 2002; Corradini *et al.* 2001). Such samples can be created by controlled mechanical disruption of samples of the same product by simply stirring them in different ways.

Preliminary results indicated that the consistency measured as the overall resistance to flow or deformation is most probably the dominant stimulus to the sensation of a semiliquid food's consistency, which is also expressed in sensory terms like "viscosity," "thickness," "body," etc. Although yet to be employed in elucidating the relationship between microstructure and texture, squeezing flow viscometry might become a very useful tool in such an endeavor. Advances in the studies of the relation between perceived consistency, specific microstructural features of foods and their rheological properties would have to rely on simultaneous progress in structure characterization (e.g., Aguilera and Stanley 1999; Sun *et al.* 2001) and on better understanding of the biophysical mechanisms that are involved in the sensation that we call "consistency."

ACKNOWLEDGMENTS

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