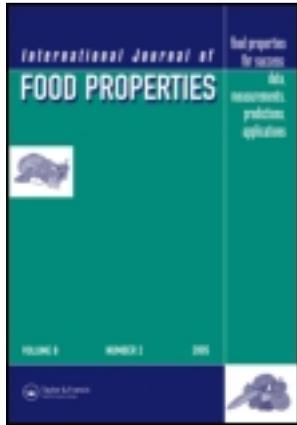


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### Yield Stress in Foods: Measurements and Applications

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## YIELD STRESS IN FOODS: MEASUREMENTS AND APPLICATIONS

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*Though the presence of true yield stress has been debated, it has been accepted as an engineering reality. Now, yield stress is routinely measured and used in the food industry not only for basic process calculations and manufacturing practices, but also as a test for sensory and quality indices and to determine the effect of composition and manufacturing procedures on structural and functional properties. In this article, we present a comprehensive review of various measurement methods and the applications of yield stress in many practical situations. The measurement methods have been grouped under traditional and recent methods. An emphasis was placed on the development of measurement methods applied to a variety of foods, and the use of yield stress as a parameter to evaluate certain food material attributes: flow behavior, end-use quality, processability, composition, and so on.*

### INTRODUCTION

Yield stress is a well recognized physical and rheological property for liquid and solid materials. The yield stress is classically defined as the minimum shear stress that must be applied to the material to initiate flow.<sup>[1]</sup> Materials that exhibit yield stress are typically multiphase systems such as cements, soils, paints, pastes, printing inks, greases, and a variety of food products such as salad dressings, sauces, and spreads.

The idea of yield stress was introduced in the 1920s by the work of Bingham, Schwedoff, and Green, and since then, its concept and definition have been continuously debated, especially after Barnes and Walters<sup>[2]</sup> challenged its existence in their article “The Yield Stress Myth?” The controversy hinges on the early statements describing solid materials as those exhibiting “no continuous deformation (or flow) upon decreasing stresses below the yield stress value.” The debate continued thereafter with the works by Evans,<sup>[3]</sup> Astarita,<sup>[4]</sup> Schurz,<sup>[5]</sup> and Spaans and Williams.<sup>[6]</sup> The existence of yield stress in fluids still remains controversial; however, there is much to validate that yield stress is an “engineering reality”.<sup>[7]</sup> For instance, flocculated suspensions and dispersions usually exhibit yield stress as individual particles tend to interact and aggregate resulting in flocs and consequently form a three-dimensional network structure.<sup>[8,9]</sup> Therefore, yield stress may also relate to the strength of the coherent network structure throughout the volume of the material as the force per unit area required to break down the structure.<sup>[1,8,10,11]</sup>

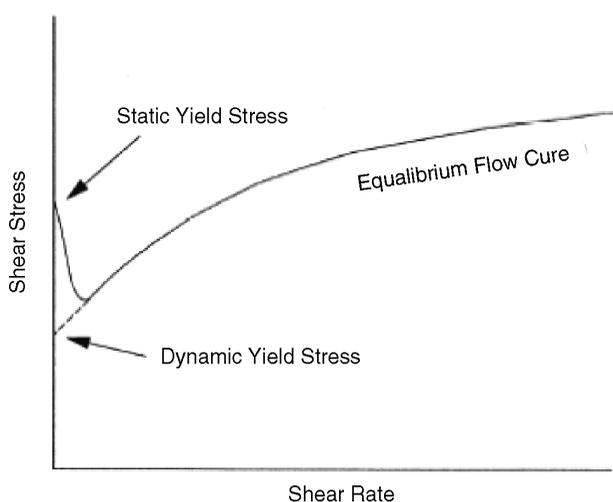
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In addition, yield stress is generally denoted as the transition stress at which a material behaves either as elastic solid-like or viscous liquid-like. This transition usually occurs within a range of stresses, in which the material exhibits viscoelastic behavior.<sup>[1]</sup> This transition behavior may be distinguished based on the Deborah number ( $De$ ), which is defined as the ratio of the characteristic time of the material to the characteristic time of the deformation process. Thus, a material can behave as either a solid-like at high  $De$  (i.e., elastic deformation with short characteristic time) or liquid-like at low  $De$  (i.e., viscous flow with long characteristic time). Consequently, the timeframe of yield measurement is also a key parameter.<sup>[12]</sup> Cheng<sup>[12]</sup> also suggested that the magnitude of the measured yield values is closely related to creep, stress growth, thixotropic breakdown and recovery, and characteristic times of the transient responses. These controversial debates notwithstanding, the quantitative knowledge of yield stress is used extensively in diverse areas of study—colloidal science, food engineering, biomedical sciences, and geology. The idea of yield stress is now a well accepted physical reality. However, as cautioned by Barnes,<sup>[9]</sup> defining yield stress and using mathematical models to characterize material behavior should only be used over an appropriate range of shear rates and under proper applicability.

In the food industry, the definition of yield stress as the minimum shear stress required to initiate flow,<sup>[8,13,14]</sup> is generally accepted due to the limited time-scale of most food processing operations (e.g., emptying a vat) and other food-related activities (e.g., eating).<sup>[9]</sup> The idea of yield stress as the transition stress and as a measure of the structural strength of food dispersions like mayonnaise and tomato puree are also well known.<sup>[11,15,16,17]</sup> Cheng<sup>[12]</sup> introduced the concept of static and dynamic yield stresses, which is useful for food processing applications (Fig. 1). The former represents the yield stress of an undisturbed sample; and the latter, a broken down sample. In general, the magnitude of static yield stress is significantly higher than that of the dynamic yield stress. This concept is important considering that most foods exhibit time-dependent rheological properties and undergo physicochemical changes during storage and/or processing.<sup>[14,15]</sup>

A precise, quantitative understanding of yield stress in foods is important for several practical applications—from process calculations to product development. For example,



**Figure 1** Static and dynamic yield stress (from<sup>[15]</sup>).

the knowledge of yield stress is helpful in estimating how well a fluid food, such as tomato ketchup, drains from a bottle.<sup>[18]</sup> There are several other applications of yield stress in foods: thickness of coating layer (chocolate on ice cream bar, glazing on doughnuts and cakes), settling of particles in fluids (spices in salad dressing, chocolate in chocolate milk), spreadability (cream cheese, mayonnaise), mouthfeel (creaminess of yoghurt), and process design calculations (flow and velocity profiles through pipelines).

## MEASURING YIELD STRESS IN FOODS

The most common method to obtain yield stress is to extrapolate the shear stress versus shear rate curve back to zero shear rate. However, extrapolated yield stress values are strongly influenced by the rheological model chosen.<sup>[15, 19]</sup> An alternative to this is to measure yield stress directly, which relies on an independent assessment of the stress at which the material starts to flow.<sup>[11]</sup> Direct measurements with conventional rheometers involve testing a material with creep/recovery, stress relaxation, and stress growth techniques. Other methods such as cone penetration, vane method, and dynamic oscillatory measurements have also been studied.<sup>[8, 12, 13, 20]</sup> A modified squeezing flow apparatus,<sup>[21]</sup> slump tests,<sup>[22]</sup> and measurements of static yield stress of suspensions using a slotted-plate device,<sup>[23]</sup> are among the recently developed methods.

Two major problems encountered in the determination of the yield stress lie on the reproducibility of the experimental data and on the vastly different yield values obtained from different techniques.<sup>[15]</sup> As indicated by Cheng,<sup>[12]</sup> yield stress is a time-dependent property; hence, a proper time scale of the experiment must be selected to minimize discrepancy among measurements. Furthermore, most suspensions show thixotropic characteristics, which cause development of a more ordered structure or structural recovery if the applied strain rate is small (i.e., long measurement time).<sup>[19]</sup>

To make matters worse, fluids that exhibit yield stress are often more difficult to measure and the type of viscometer may impose additional limitations due to possible wall slip.<sup>[24]</sup> These effects are associated with the displacement of the dispersed phase away from solid boundaries due to surface, hydrodynamic, viscoelastic, chemical, and gravitational forces. As a result, the liquid near the walls are depleted of particles and have lower viscosity, leading to higher measured shear rates at fixed shear stresses.<sup>[19]</sup> Improvements to existing methods and new methods have been developed to obtain reliable and precise yield stress measurements in foods.

### Traditional and Common Methods

**Extrapolation.** Extrapolation of the shear stress versus shear rate data obtained from conventional rheometers is the most common indirect technique to measure yield stress. The experimental data, also referred to as equilibrium flow curves, can be interpreted with or without a rheological model such as the Bingham, the Herchel-Bulkley or the Casson. Extrapolating the experimental data back to zero shear rate and obtaining the yield stress value at the shear stress intercept is an easy and quick procedure; however, it can only be done for linear data.<sup>[24]</sup> An alternative numerical technique is to plot apparent viscosity ( $\eta_a$ ) versus shear stress and determine the yield value at the point where  $\eta_a$  tends to infinity.<sup>[15]</sup> Similarly, Nguyen and Boger<sup>[24]</sup> pointed out that a slope of  $-1$  for a logarithm plot of  $\eta_a$  versus shear rate curve over a range of low shear rate often indicates the presence of yield stress (e.g., Bingham fluids). The use of rheological

models is helpful for proper extrapolation. However, it is important to reiterate that the selection of a particular model depends on the shear rate range and on the intended application.<sup>[9,24]</sup>

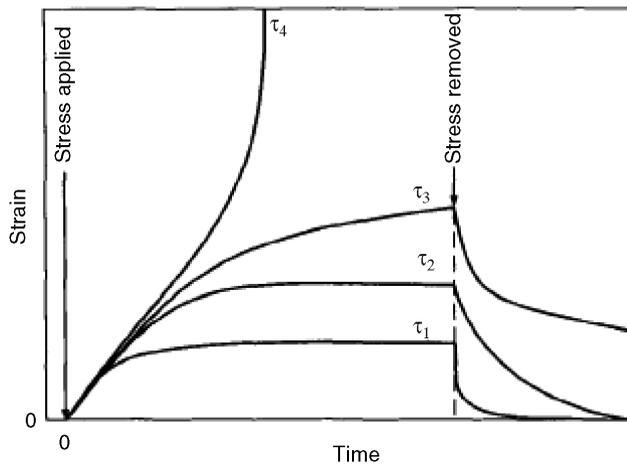
Overall, the goodness of the extrapolated yield stress value relies on the accuracy of the experimental data in the low shear rate range. In addition, as mentioned earlier, wall slip effects, fracture, and expulsion of the fluid sample are some of the problems encountered while using conventional rheometers, which may affect not only the extrapolated yield stress value, but also the rheological flow behavior. Nguyen and Boger<sup>[24]</sup> described in detail the main features and problems with most common types of viscometers, and proposed some methods to correct these problems. However, the yield stress obtained from extrapolated technique cannot be regarded as an absolute material property or “true yield stress”.<sup>[24]</sup> The extrapolated yield stress is often used as a quick estimate and to compare results from other methods.

**Stress relaxation test.** In this method, the fluid material is first sheared at either constant shear rate or constant shear stress in a conventional rotational viscometer, followed by bringing the material to rest either gradually or suddenly. The yield stress, also called as the relaxed stress, is then measured as the limiting or residual stress remaining in the fluid upon cessation of flow.<sup>[11]</sup> During the test, it is important to ensure that the initial applied stress is higher than the yield stress. When the shear stress in the relaxed state decays to a constant level it represents the yield value. This method has been primarily applied to study linear viscoelasticity of food materials, with little work done for yield stress measurements. One of the reasons is the pre-shearing required, which damages the material structure. Therefore, stress relaxation technique is suitable only for fluids whose yield stress is not dependent on shear history.<sup>[24]</sup>

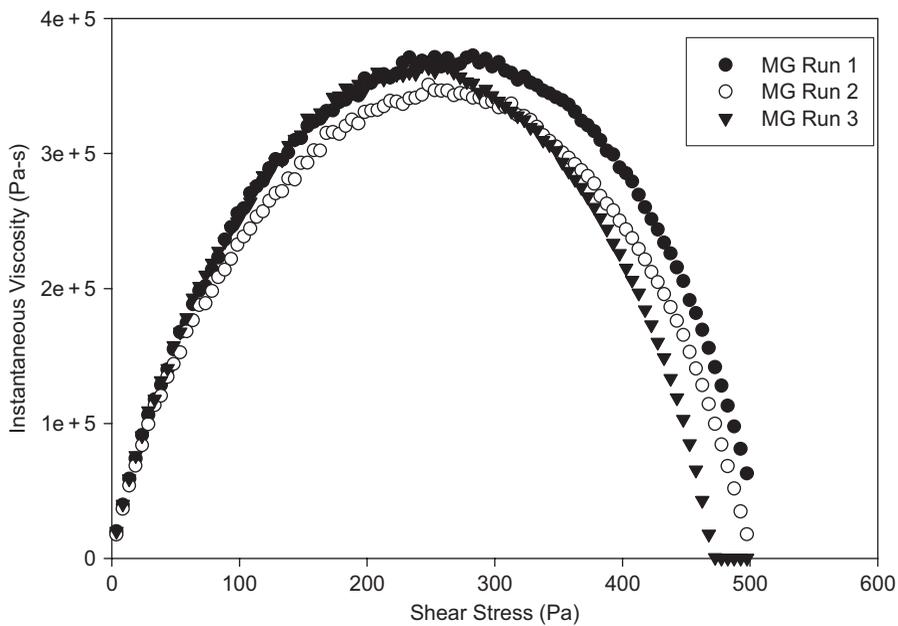
**Creep/recovery response.** The creep/recovery response consists of applying a constant shear stress in steps to the fluid material as illustrated in Fig. 2. If stresses applied are below the yield stress, the test material behaves as an elastic solid with a complete recovery upon removal of stress and it will not flow. This is observed by a horizontal curve with slope nearly zero ( $\tau_1$  and  $\tau_2$ ). When the applied stresses are greater than the yield value, the deformation or strain increases indefinitely with time and viscous flow ensues ( $\tau_4$ ). The yielding point can be detected from a drastic change of slope in the time vs. shear strain curve; sometimes, it is difficult to identify the critical shear stress as it approaches the yield stress. It can be seen in Fig. 2 that the yield stress lies between  $\tau_2$  and  $\tau_3$ . Overall, creep/recovery test is a more sensitive and less destructive than other methods for measuring yield stress. However, measurement duration can be very long to determine the steady creep compliance; influence the accuracy of the yield values due to shear history and thixotropic; and possibly cause some physicochemical effects.

**Shear Stress Ramp Test.** This method is an alternative to creep/recovery technique to shorten the measurement duration. The test is performed by applying gradually increasing stresses onto the sample. The instantaneous (or apparent) viscosity is monitored for the presence of an inflexion point, which indicates onset of flow, and the yield stress is given at the corresponding shear stress (Fig. 3). The measurement of the stress ramp curves are often performed with parallel plate or cone-plate geometries. Possible errors involved are slipping of the sample and effects due to gap size.

**Cone Penetrometer.** Cone penetration is a popular technique used for testing strength properties of soils and other materials of high consistency. In this test, a metal cone of specific dimensions (angle  $\alpha$  and height ( $H$ ) and weight ( $W$ ) is forced into a flat

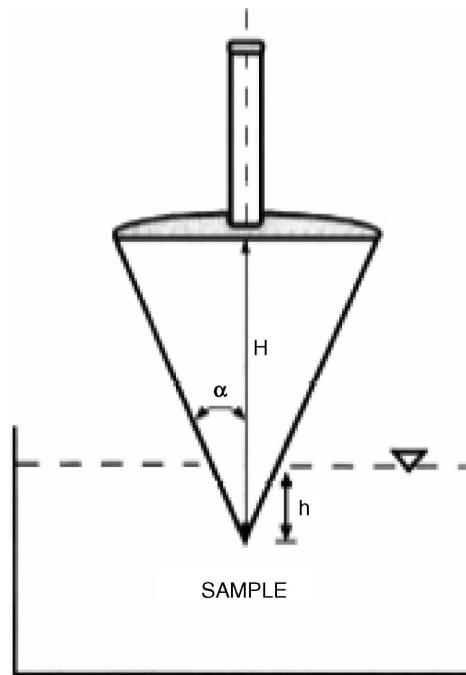


**Figure 2** Typical strain-time curves obtained during yield stress measurement by the creep-recovery test (from<sup>[1]</sup>).



**Figure 3** Stress ramp curve for margarine (from<sup>[21]</sup>).

and smooth horizontal surface of test specimen (Fig. 4). The penetration can be performed in three modes of operation<sup>[25]</sup>: (1) a metal cone is let to sink and the depth of penetration is measured after a fixed time; (2) a cone is released into the sample and the depth of penetration is measured when the cone is at rest due to the yield stress of the material; and (3) a cone is pushed into the sample at a constant speed and the force required for the penetration is recorded. In the constant force mode, the cone penetrates quickly at first, but it



**Figure 4** Illustration of a cone penetrometer assembly (from<sup>[25]</sup>).

gradually slow down until it comes to rest. The “apparent yield stress” ( $\sigma_{app}$ ) is calculated as a function of the penetration depth  $h$  when the cone stops<sup>[25]</sup>:

$$\sigma_{app} = \frac{W}{\pi h^2 \tan^2(\alpha)}. \quad (1)$$

The cone penetration is considered an empirical method for rapid evaluation of consistency of a variety of solid and semi-solid foods. In fact, this method has been employed extensively for measuring the stiffness of dairy products.<sup>[24]</sup> As for measuring the yield stress, the method is also considered suitable for fluids with high yield stress and low viscosities as the viscous resistance during cone penetration is minimal. Nguyen and Boger<sup>[24]</sup> points out that viscous effects retard cone penetration resulting in over estimation of the yield value. On the other hand, it is disadvantageous when measuring highly viscous materials, such as butter, since it might take long time for the cone to reach an equilibrium condition on its own weight.

Other methods based on similar principle of forcing an object through the material under test have been investigated. For example, the falling needle viscometer, which is used to measure the steady shear viscosity of Newtonian and non-Newtonian viscoelastic fluids, has been attempted for yield stress measurements.<sup>[26]</sup> This method can provide an indirect measure of yield stress, once the density difference between the needle and the liquid is corrected for. However, the needle is not stationary in the liquid.

Uhlherr et al.<sup>[27]</sup> proposed a new method for static measurement of yield stress using a cylindrical penetrometer. The principle is similar to that of the falling needle

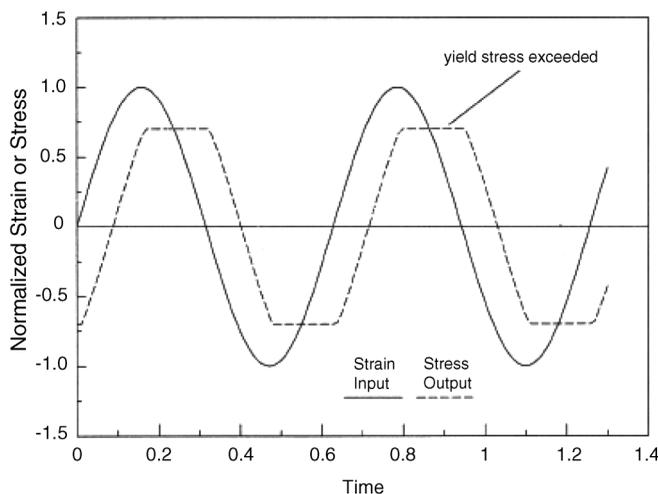
viscometer—the penetrometer falls under gravity toward an equilibrium position after being gently released in a fluid. Once the penetrometer is partially immersed, it reaches static equilibrium at the position where the yield stress is achieved. The yield values measured from this technique are comparable to those values obtained from other tests such as vane, extrapolation, stress ramp, and creep for Carbopol and titanium dioxide solutions.

**Dynamic oscillatory testing.** Dynamic mechanical measurement is an alternative method for the determination of yield stress as it provides useful information about the structural and deformation behavior of viscoplastic fluids at below or near the yield stress.<sup>[24]</sup> During oscillatory testing, the material is subjected to a sinusoidal strain and the resulting stress is measured as a function of both time and frequency. Depending on the strain amplitude ( $\gamma_o$ ), small deformations occur within the linear viscoelastic region, whereas large deformations will be in the non-linear region.

Dynamic measurements in the linear viscoelastic region of multiphase and composite materials exhibit a low frequency plateau, where the linear viscoelastic terms are independent of frequency.<sup>[24]</sup> The presence of this plateau has been suggested to be due to the existence of a network structure, which can be correlated to the yield stress (Fig. 5), whereas plateaus are absent for non-yield stress fluids.<sup>[24]</sup>

Dynamic measurements are not popularly used for yield stress determination, because it is difficult to achieve the extremely small amplitude required so that the stress and strain sinusoidal waveforms are completely in phase.<sup>[1]</sup> On the other hand, it might be an advantageous procedure as the small deformation causes minimum disturbances to the structure of the fluid material.<sup>[24]</sup> Furthermore, Liddell and Boger<sup>[1]</sup> pointed out that measurement in the non-linear viscoelastic region can yield superior results, once the plateau where the non-linear viscoelastic functions are independent of frequency.

**Static stability on an inclined plate.** Nguyen and Boger<sup>[24]</sup> described a technique to measure the yield stress of viscoplastic fluids on an inclined plate. This method takes into consideration the shear stress of the fluid perpendicular to an



**Figure 5** Dynamic oscillatory measurement (from<sup>[15]</sup>).

inclined plane of angle  $\alpha$  at a position  $y$  measured from the free surface end. This relation is given as<sup>[24]</sup>:

$$\tau(y) = \rho \cdot g \cdot h \cdot \sin \alpha, \quad (2)$$

where,  $\rho$  is the density of the fluid material and  $g$  is the acceleration of gravity. Under the above conditions, flow only occurs when shear stress is greater than the yield stress  $\tau(y) > \tau_o$ . The yield stress is determined by placing a uniform fluid layer on an initially horizontal plane, followed by progressively increasing the angle of inclination of the plane until a critical value is reached, whereby the fluid starts to flow (Fig. 6). An alternative method is to keep the angle of inclination  $\alpha$  fixed and measure the final fluid depth when flow stops after drainage. Although these techniques seem to be relatively simple, accuracy of the measurements is strongly influenced by the precision in measuring the critical angle and the fluid thickness of the.

A very similar but different method has also been attempted—withdrawing a plate immersed in a fluid bath at constant speed and measuring the thickness of the material adhering to the plate (Fig. 7). The plate remains in contact with the liquid bath throughout the process, and the amount of material coated to the plate is related to the yield stress.

Lang and Rha<sup>[30]</sup> further investigated this method and concluded that the yield stress determined from the thickness of the fluid coated on the vertical plate is not comparable to those obtained with other techniques. De Kee et al.<sup>[29]</sup> examined the post-withdrawal drainage behavior of Newtonian and viscoelastic fluids and viscoelastic suspensions with a computer-driven laser interferometry, which was used to investigate the transient film thickness. Their results also indicated that the plate-withdrawing technique ineffective for calculating yield stress. Some problems encountered were related to interfacial

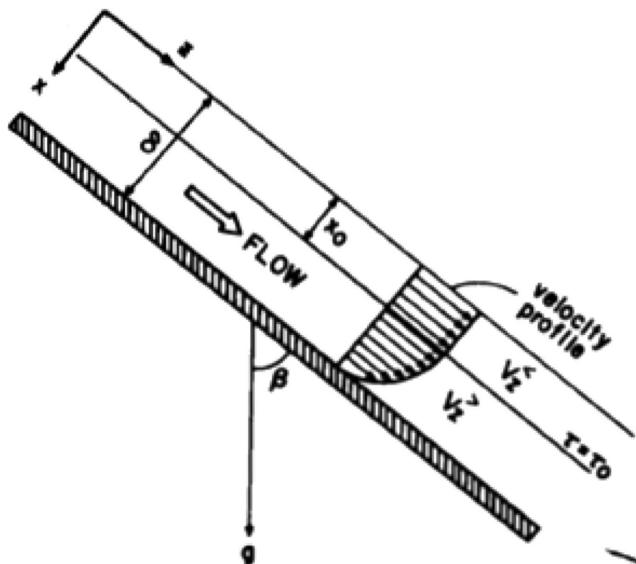


Figure 6 Schematic of the flow in an inclined plate (from<sup>[28]</sup>).

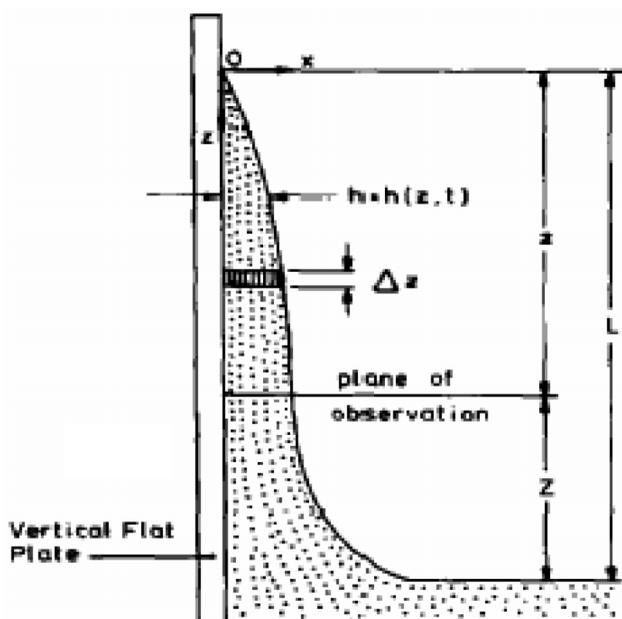


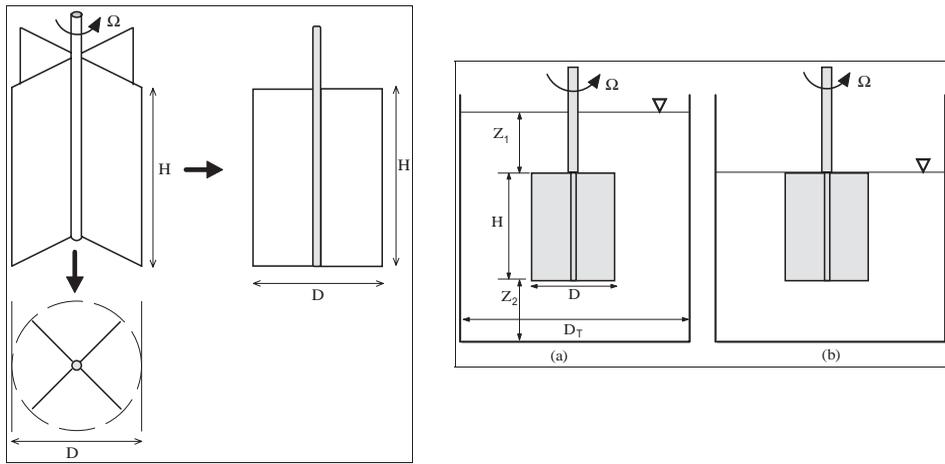
Figure 7 Illustration of post-withdrawal drainage process (from<sup>[29]</sup>).

interactions, rate of withdrawal from the fluid, and draining time of material from the plate.<sup>[14]</sup>

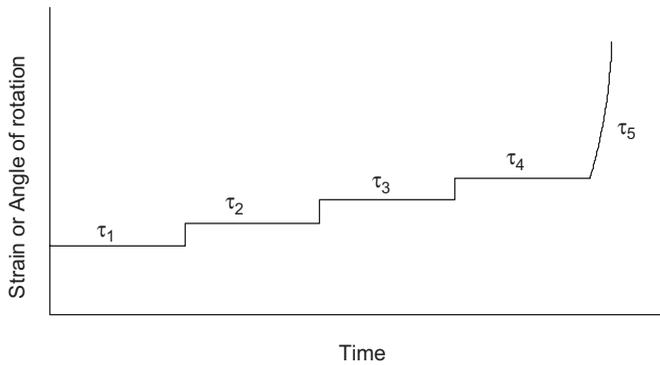
**Vane technique.** The vane method was originally employed to eliminate slipping of the sample while studying the coagulation of sodium clay gels<sup>[31]</sup>; it was further developed to measure flow properties and shear strength of soils.<sup>[8,31,32]</sup> The vane geometry consists of a vane spindle with typically four to eight thin blades arranged at equal angles, centered on a narrow cylindrical shaft of a rheometer or viscometer. Figure 8 shows a schematic of a typical four-bladed vane and how it is used during measurement.

The vane can be operated under controlled shear-rate mode or controlled shear-stress mode; in both cases the yield stress is the minimum stress required for continuous rotation of the vane. In the controlled shear-stress mode, a constant stress is applied in steps to the vane immersed in the sample. The resulting data corresponds to the strain (or angle of rotation) as a function of time for each increased stress step. The yield stress is determined as the stress corresponding to the point at which there is a rapid increase in the creep angle or strain for example between  $\tau_4$  and  $\tau_5$  in Fig. 9. Vane method also minimally disturbs the sample upon insertion of the vane spindle, which is critical when measuring weakly and delicately structured materials such as foams and emulsions.<sup>[8,25,31,32]</sup> Sample disturbance may also be minimized by using the original product containers such as for foods.<sup>[33]</sup> The vane method has been suggested as a standard method for determining the yield stress of foods.<sup>[34]</sup>

In the controlled shear-rate mode, a constant rotational speed is applied to the vane, and the resulting torque is measured as a function of time (Fig. 10). Studies have shown that rotational speeds between 0.1 and 8 rpm produce superior results, and yield stress values measured were essentially constant.<sup>[14]</sup> The yield stress can therefore be obtained



**Figure 8** Schematic of a typical four-bladed vane (left) and how it is used during measurement (right). [ $H$  = vane height,  $D$  = vane diameter,  $D_T$  = container diameter,  $\Omega$  = angular velocity, (a): immersed in the sample ( $Z_1$  and  $Z_2$  height of sample above and below the vane, respectively); (b): top surface in level with sample] (from<sup>[25]</sup>).

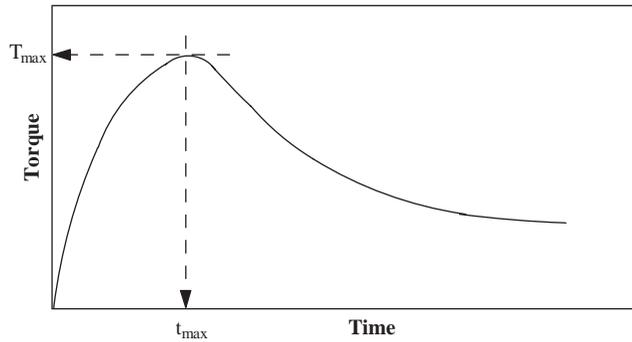


**Figure 9** Typical response curve for yield stress measurement with the vane method under controlled shear-stress mode. Shear stress  $\tau_1$  through  $\tau_5$  are applied each for certain duration.

through a quick single-point determination, based on the total torque to overcome the yield stress ( $\sigma_o$ ) of the fluid. A simplified equation is given as:

$$\sigma_o = \frac{2T_{max}}{\pi d^3} \left( \frac{h}{d} + \frac{1}{m+3} \right)^{-1}, \tag{3}$$

where,  $T_{max}$  is the maximum torque;  $h$  is the height of the vane;  $d$  is the diameter circumscribed by the tip of the vane blades; and  $m$  is a constant. Two key assumptions are made: 1) the shear stress is uniformly distributed over the end surfaces of the vane; and 2) the material yields along a cylindrical surface outlined by the edges of the vane blades. If the first assumption is true, then  $m = 0$ . Steffe<sup>[14]</sup> showed that errors can be up to nearly 15% if this assumption is violated.



**Figure 10** Typical torque versus time response curve for yield stress measurement with the vane method under controlled shear-rate mode (from<sup>[25]</sup>).

An alternative solution to eliminate errors associated to the non-uniformity of stress distribution over the vane ends is to measure the response when the top of the vane blades are aligned evenly with the top surface of the sample (i.e.,  $Z_1 = 0$ , see Fig. 8). Thus, the following equation can be used to calculate the yield stress:

$$\sigma_o = \frac{2T_{\max}}{\pi d^3} \left( \frac{h}{d} + \frac{1}{6} \right)^{-1} \quad (4)$$

In general, the assumption of  $m = 0$  is usually accepted<sup>[15]</sup>; thus one could rather measure the torque response when the blades are entirely immersed in the sample:

$$\sigma_o = \frac{2T_{\max}}{\pi d^3} \left( \frac{h}{d} + \frac{1}{3} \right)^{-1} \quad (5)$$

Steffe<sup>[14, 15]</sup> suggested a procedure to evaluate the validity of  $m = 0$  assumption by calculating the yield stress as the slope of  $T_{\max}$  vs.  $h$  curves, using vanes of same diameter but different lengths. With respect to the second assumption, the actual sheared surface can be up to 5% larger than the vane dimensions.<sup>[1,14]</sup> Barnes and Nguyen<sup>[31]</sup> pointed out that yield stress values may vary up to 10% if diameter of the sheared surface were greater than the circumscribed circle defined by the blade tips. Steffe<sup>[14]</sup> examined the results obtained using different vane dimensions ( $h$  and  $d$ ) and container diameter ( $D$ ) and recommended the following:  $1.5 \leq h/d \leq 4.0$ ;  $D/d > 2.0$ ;  $Z_1 = 0$  or  $Z_1/d > 1.0$  if the vane is fully immersed in the sample; and  $Z_2/d > 0.5$ . Genovesse and Rao<sup>[11]</sup> used the vane technique to determine the static and dynamic yield stresses of six commercial foods. The procedure used was to measure the peak torque at undisrupted conditions (static yield stress) followed by applying a continuous cyclical rotation to disrupt the sample and measuring the peak torque afterwards (dynamic yield stress).

### Recent and Improved Methods

**Squeezing flow.** Squeezing flow has been primarily used to measure the flow properties of highly viscous materials such as polymer melts. These flow arrangements

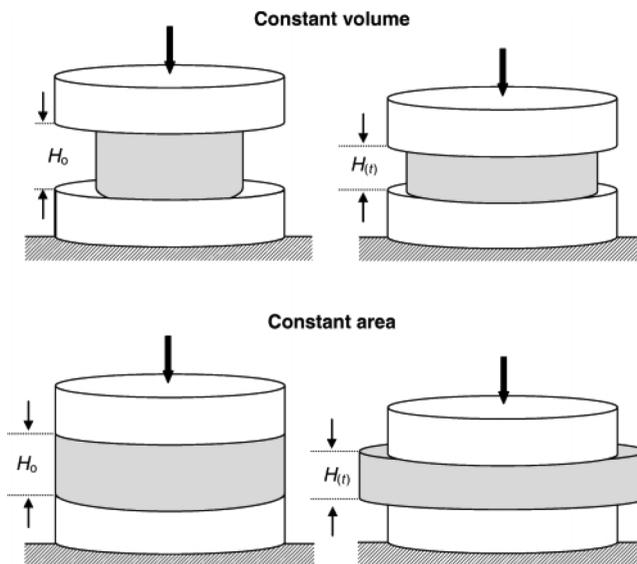
were originally tested with industrial viscometers known as “parallel plate plastometers”.<sup>[35]</sup> Since then, the technique has been applied for rheological measurements of a wide range of materials, including purely viscous liquids, yield stress fluids, viscoelastic solids and liquids, and purely elastic solids.<sup>[36]</sup> Introduced to foods in the mid-1980s, squeezing flow has been a useful method to measure the rheological properties of semi-liquid and semi-solid food materials such as processed and melted cheeses, butter, dough, peanut butter, etc.<sup>[25,27]</sup>

The method has become very attractive for measuring rheological properties of very viscous materials and fluids with yield stress because it minimizes the two major problems encountered with conventional rotational viscometers: slip and extensive structural disruption upon insertion of sample into narrow viscometer gap.<sup>[38]</sup> In addition, the squeeze flow geometry allows producing a wide range of strains and internal deformations of the material.<sup>[36]</sup>

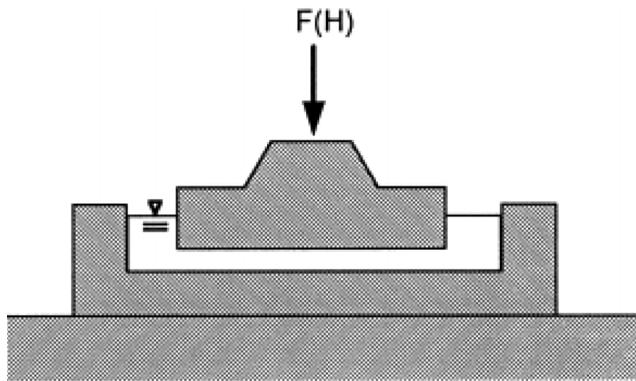
There are basically three test geometries (Figs. 11 and 12): 1) specimens with a constant area and changing volume; 2) specimens with constant volume and changing area, and 3) “imperfect squeezing-flow.” In the first geometry, the specimen radius is usually equal to that of the plates, which guarantees a known contact area at all time and minimization of errors. However, as the sample flows and accumulates outside the plates during compression, an additional variable, pressure that is difficult to predict, is created.<sup>[36]</sup>

In the constant volume geometry, the plates are larger than the specimen and the radial interface is free. Engmann et al.<sup>[36]</sup> points out that stresses at the sample edges are more clearly defined, reducing additional pressure build-up at the edges. In imperfect squeezing flow, the bottom plate is replaced by a shallow container.<sup>[39]</sup> However, not only the boundary conditions at the edges of the plates are unknown, the contact areas between plates and fluid of different size can cause an asymmetric flow.<sup>[36]</sup>

The flow pattern during the squeezing of a fluid can be classified as being lubricated (perfect slip) and unlubricated (frictional or no slip). Lubrication of the parallel plates can



**Figure 11** Geometry of squeezing flow test under constant area and constant volume (from<sup>[38]</sup>).



**Figure 12** Schematic of imperfect squeezing flow apparatus (from<sup>[40]</sup>).

be achieved intentionally (e.g., use of paraffin oil) or by the specimen itself (e.g., peanut butter and melted cheese). The flow pattern can be distinguished by noticing whether the expelled fluid is flat (lubricated) or rounded (non-lubricated). The adhesion and friction between the sample and the plates creates a barrier effect resulting in shear flow, and consequently, during frictional squeezing flow, squeezed specimen is both elongated and sheared. The flow behavior for lubricated, non-lubricated, and partially lubricated conditions have been theoretically analyzed and reviewed by Engmann et al.<sup>[36]</sup>

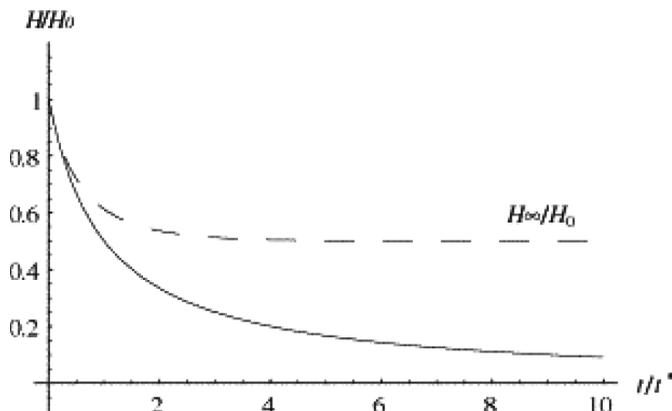
Another factor that makes squeezing flow an attractive method is its simplicity. The technique can be performed in two forms: 1) constant or controlled deformation rate; and 2) constant force or stress. At constant deformation rate, the cylindrical specimen is compressed between two parallel plates at constant speed, whereby the force-time or force-height is measured. On the other hand, in constant stress, a creep test is performed and the response is a height-time relationship.

The determination of the yield stress from squeezing flow is based on the residual stress after shear and relaxation. The evaluation takes into consideration that a viscous-plastic fluid (i.e., Bingham plastic and Herschel-Bulkley fluid) when subjected to constant compressive stress flows until it reaches an equilibrium height ( $H_L$ ) as shown in Fig. 13. The yield stress obtained from the squeezing flow rheometry is sometimes referred to as the apparent extensional yield stress<sup>[41]</sup> or the dynamic yield stress.<sup>[36]</sup>

Campanella and Peleg<sup>[42]</sup> evaluated the yield stress of tomato ketchup, mustard and mayonnaise with a non-lubricated squeezing flow apparatus under constant force and constant area using the following analytical expression:

$$\sigma_o = \frac{3.W.H_L}{2\pi.R_o^3}, \quad (6)$$

where,  $W$  is the load applied on the upper plate;  $H_L$  the equilibrium height; and  $R_o$  is the initial radius of the cylindrical specimen. As for tests under lubricated squeezing flow (LSF), Engmann et al.<sup>[36]</sup> points out that dynamic yield stresses cannot be obtained under constant area conditions due to the fact that the material will either continue to deform indefinitely or not deform at all.



**Figure 13** Typical profile in squeeze flow testing for two types of fluids: Newtonian (full line) and Bingham (dashed line) (from<sup>[36]</sup>).

Yang<sup>[43]</sup> presented an expression for LSF of Herschel-Bulkley fluids relating the squeezing force and the fluid thickness. Based on this Ak and Gunasekaran<sup>[41]</sup> derived an alternative analytical equation to determine the apparent extensional yield stress of a Herschel-Bulkley fluid under constant force and constant volume:

$$\sigma_o = \frac{W.H_L}{\pi.R_o^2.H_o}, \quad (7)$$

where,  $H_o$  is the initial height of the specimen. While yield stress measurements of semi-liquid foods by squeezing flow are often difficult to perform at constant deformation rate, an estimate of the yield stress can be obtained by determining the residual apparent yield stress with imperfect squeezing flow. Suwonsichon and Peleg<sup>[40]</sup> evaluated this technique, in which the force exerted by the fluid (the residual force or stress) correlates to the yield stress value. For fluids without yield stress, the recorded force will drop to the buoyancy force instead to zero.

Moreover, an alternative to controlled deformation rate and imperfect squeezing flow geometries is the creep test. Sun<sup>[21]</sup> measured the yield stress of concentrated spreadable foods using a modified squeezing-flow apparatus, known as the UW Meltmeter, which has been developed and used to measure the melt and flow behavior of cheeses by Gunasekaran and his co-workers.<sup>[44]</sup> This device consists of one or two sample wells (7-mm deep and 30-mm in diameter) with an inner and outer ring, which makes it easier during sample loading. A stationary center piston allows the outer ring to move up and down through a lever, whereby when it is at the lower position, a cylindrical sample specimen is formed taking the shape and dimensions of the sample well (Fig. 14).

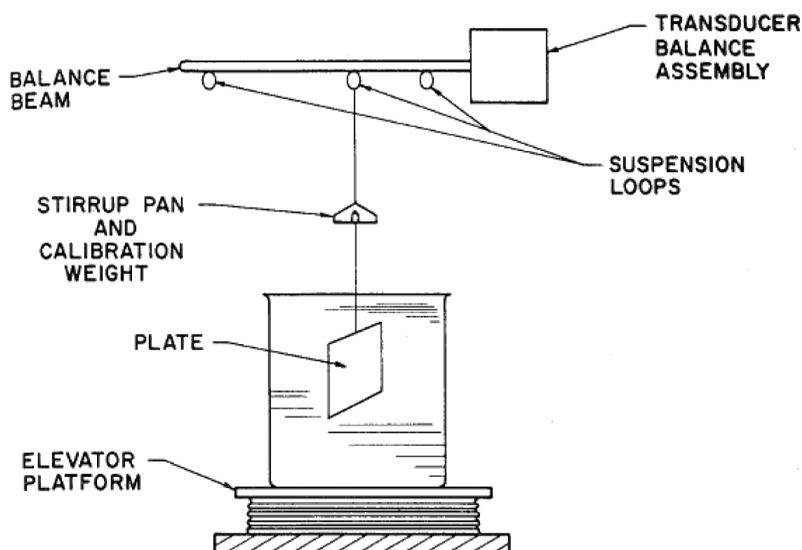
The UW Meltmeter performs squeezing flow test under constant force, constant volume and changing area. Store-bought peanut butter, margarine and mayonnaise were used in this study due to their textural and subjective spreadability (semi-hard, mild and easy, respectively). The “apparent extensional yield stress” of these selected spreadable foods were determined by applying the Herschel-Bulkley model proposed by Ak and Gunasekaran,<sup>[41]</sup> shown in Equation 7. The yield stress values measured with the UW Meltmeter and vane technique were in good agreement for all three spreadable food products.



**Figure 14** Picture of the dual sample well UW Meltmeter (Left) and sample formed with the lever in the down position (from<sup>[21]</sup>).

Overall, the UW Meltmeter is a simple, quick, and reliable technique to measure the yield stress parameter of a variety of semi-liquid foods, including spreadable foods.

**Plate method and slotted-plate device.** The plate method was developed to overcome some of the problems associated with the assumptions of the vane method that are usually overlooked<sup>[19,28]</sup>: sample yields along the cylinder described by the blades and uniform stress distribution along the blades. The plate method was specifically designed to produce a uniform stress distribution on the plate surface and to be applicable for both high and low concentration suspensions. De Kee et al.<sup>[28]</sup> used this static method to determine the yield stress of several liquid and semi-liquid foods (condensed milk, corn syrup, molasses, ketchup, mayonnaise and tomato paste). The experimental procedures consist of immersing the plate into the glass container filled with sample material (Fig. 15). The platform is then lowered at controlled and constant speed (from 0.0127 to



**Figure 15** Illustration of the plate method in measuring yield stress (from<sup>[28]</sup>).

2.54 cm/min). Initially, the yield stress fluid remains fixed to the plate and as the platform descends a force is exerted on the balance beam causing it to pivot.

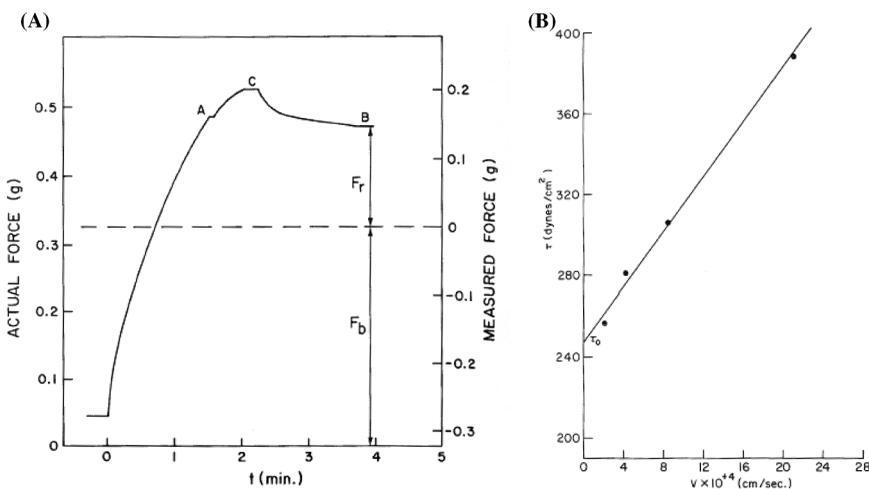
This stage is represented between time zero and point A in the graphical response shown in Fig. 16A. Once the fluid starts moving with respect to the plate, the force acting on the plate surface exceeds the yield stress (shown by the change in slope after point A), following an increase in force to a steady-state value for a given rate of descent (point C). Point B represents the stage in which the platform stops and the balance beam cannot return to its original horizontal position due to the yield stress. The yield stress is then determined as<sup>[28]</sup>:

$$\tau_o = \frac{(F_b + F_r)g}{s}, \tag{8}$$

where,  $F_b$  is the buoyant force (g);  $F_r$  is the recording force at point B (g);  $g$  is the acceleration due to gravity (cm/s<sup>2</sup>); and  $s$  is the plate surface area from both sides and without the edges (cm<sup>2</sup>).

An alternative procedure is to extrapolate the yield stress value from a shear stress vs platform speed curve (Fig. 16B). Although, the yield stress for the liquid and semi-liquid foods measured using the plate apparatus agreed closely with values determined from other methods, different suspensions and the type of plate used may cause discrepancy among the results, not to mention that yielding which may occur at the plate-sample interface as opposed to within the sample.<sup>[19,45]</sup>

As a result, slotted-plate device has been developed to combine the advantages of both the vane and the plate methods, while providing a uniform stress distribution at the plate surface and eliminating the wall effect.<sup>[45]</sup> This new device is similar to the plate method apparatus with a thin stainless steel wire connecting the slotted plate with a balance (Fig. 17A). A platform holds a double-walled beaker and a computer controls the speed of the platform. The slotted-plate device is housed in a Plexiglas box; two infrared lamps maintain constant air temperature.



**Figure 16** (A) Typical response of the plate method; and (B) yield stress obtained from extrapolation of measured shear stress versus platform speed (from<sup>[28]</sup>).

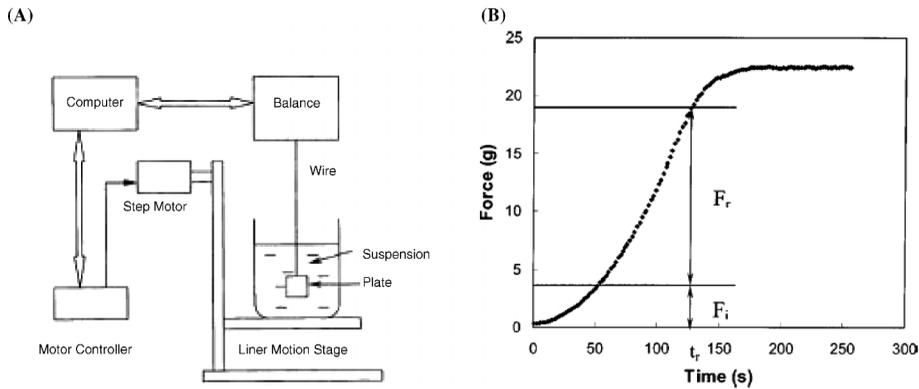


Figure 17 (A) Schematic diagram of the slotted-plate device; and (B) typical time response curve (from<sup>[45]</sup>).

The concept of the slotted-plate device lies in the fact that slots are filled once the slotted-plate is inserted into the test fluid. As the platform is lowered, the fluid exerts forces on the plate, which is continuously recorded by a balance. Flow begins once the forces exceed the yield stress, characterized by a change in slope in the response data (Fig. 17B). Consequently, the apparent yield stress can be calculated as:

$$\sigma_y = \frac{F_r}{S} = \frac{F - Fi}{S}, \quad (9)$$

where,  $F$  is the measured force recorded by the balance;  $Fi$  is the initial force (weight of plate and wire without the buoyant force);  $F_r$  is the net force produced (difference between force  $F$  and  $Fi$ ); and  $S$  is the total surface area of the plate (including those of the slots). Zhu et al.<sup>[19]</sup> confirmed that the slotted-plate technique is reliable for yield stress measurement when compared to other indirect and direct methods and recommended using a slotted plate with slot ratio, the ratio between the slot area and the total plate area, greater than 0.5. The further applications of the plate and slotted-plate devices are still under development. In addition to the materials previously tested, fluid and semi-fluid foods,<sup>[28]</sup> and mineral suspensions,<sup>[19]</sup> the slotted-plate device has been used to measure the yield behavior of cohesionless powders.<sup>[23]</sup>

**Slump tests.** The slump test has been developed as a quick and easy method to measure yield stress of thick, flocculated suspensions. Traditionally used to evaluate the “workability” of fresh concrete, Pashias et al.<sup>[22]</sup> called it “a fifty-cent rheometer,” as it offers an inexpensive way to determine the yield stress. The test consists of filling a cylindrical mold with the test fluid and lifting the mold off to allow the material to collapse and flow under its own weight (Fig. 18). The slump height, which is the difference between the initial and final heights is measured. The analytical model relating yield stress to the slump height proposed by Murata<sup>[46]</sup> was corrected by Christensen.<sup>[47]</sup> Pashias et al.<sup>[22]</sup> adapted it for a cylindrical geometry as follows:

$$\tau'_y = \frac{1}{2} - \frac{1}{2} \sqrt{s'}, \quad (10)$$

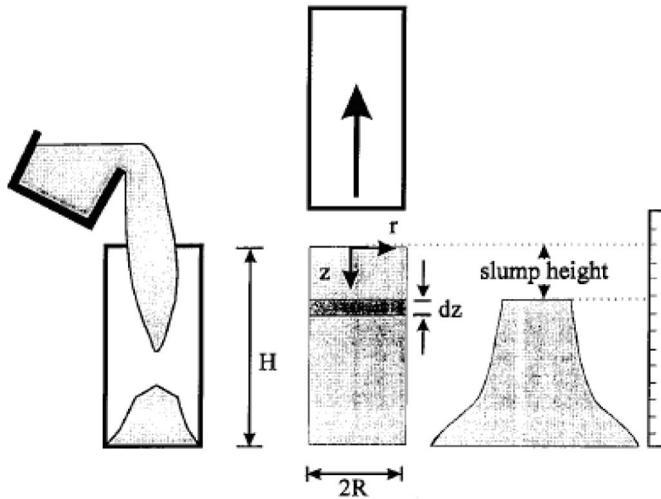


Figure 18 Schematic diagram of the initial and final state of stress distribution (from<sup>[22]</sup>).

where,  $\tau'_y$  is the dimensionless yield stress; and  $s'$  is the dimensionless slump value, which is calculated as:

$$s' = 1 - h'_0 - h'_1, \tag{11}$$

where,  $h'_0$  is the dimensionless form of the height of the non-yielded region; while  $h'_1$  is the dimensionless height of the yielded area (Fig. 19).

The standard method defined by the American Society for Testing and Materials for slump test is to use a frustum of a cone, in which the initial height is 30 cm.<sup>[48,49]</sup> With similar assumptions from Pashias et al.,<sup>[22]</sup> a relation was developed for a conical geometry<sup>[50]</sup>:

$$\tau_y, h'_0 = \frac{1}{6} \left[ (1 + h'_0) - \frac{1}{(1 + h'_0)^2} \right]. \tag{12}$$

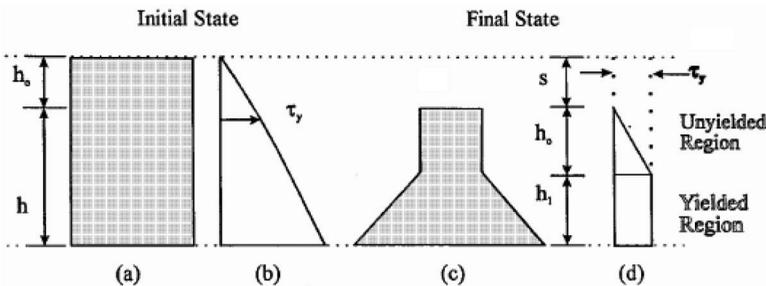


Figure 19 Schematic diagram of the slump test (from<sup>[22]</sup>).

The theory behind the analytical model shown above is explained as follow: above a critical height along the undeformed cylinder, the material experiences a shear stress that is smaller than the yield stress, and thus no flow is observed; while below this point the stress created by the weight of the material above the critical point is larger than the yield stress. After the material has slumped, flow of the material takes place in the yielded region until the cross-sectional area increases to such an extent that the stress required to support the weight reaches the yield stress. Meanwhile, the unyielded region remains intact as when the material was undeformed.<sup>[22,49]</sup> Clayton et al.<sup>[51]</sup> found that yield stress measurements of mineral suspensions were more accurate by using a cylindrical model than the conical model.

From the studies thus far, it is possible to conclude that the slump test is a robust method of measuring yield stress. It was observed that slump height is in fact dependent on yield stress, once increasing yield stress values were found for decreasing slump heights. Studies have also shown that the method is not affected by the type of flocculated material, not to mention that it is sensitive enough to distinguish between a disturbed and undisturbed material based on the slump height. Furthermore, the velocity in which the frustum is lifted off and the surface of the material does not affect measurements.<sup>[22]</sup>

A possible drawback lies in the range of yield stress values. For high yield stress materials (e.g., greater than 800 Pa), the difference between the initial and final heights is very minimal.<sup>[22]</sup> However, this problem was overcome by placing additional weights on top of the sample.<sup>[52]</sup> Omura and Steffe<sup>[48]</sup> used a centrifugal slump test to predict the yield stresses of semi-solid foods with high yield stresses. In this test, a sample is allowed to slump after it has been subjected to centrifugal force by rotating it on a circular plane. A general agreement was found between yield values of cream cheese, peanut butter, butter, margarine and Neufchatel cheese using the centrifugal slump test and those measured by the vane method. As with the conventional slump test, the centrifugal slump test is also performed by measuring slumped height and original height of the material along with the acceleration of the sample. A simplified equation is given as function of dimensionless slump height<sup>[48]</sup>:

$$\frac{z}{H} = 1 - 4(\sigma'_0)^3 + 8(\sigma'_0)^2 - 5(\sigma'_0), \quad (13)$$

where:  $z$  is slump height (m);  $H$  is original height of the sample (m);  $\sigma'_0$  = yield stress (Pa).

**Tube viscometer and magnetic imaging resonance (MRI).** The determination of yield stress of fluids using a tube viscometer is done by inducing “plug flow” through a tube. In this method, the fluid flows at constant velocity through a tube of length  $L$  and radius  $R$  under a known pressure drop ( $\Delta P$ ). The yield stress is calculated from force balance on the fluid:

$$\sigma_o = \frac{\Delta PR}{2L}. \quad (14)$$

The flow is assumed to be steady and fully developed, not to mention absent of both end and wall effects.<sup>[24]</sup> Thus, overestimation of the yield stress value are due to pressure losses as fluid enters from a reservoir and exits to open air resulting in end effects and wall

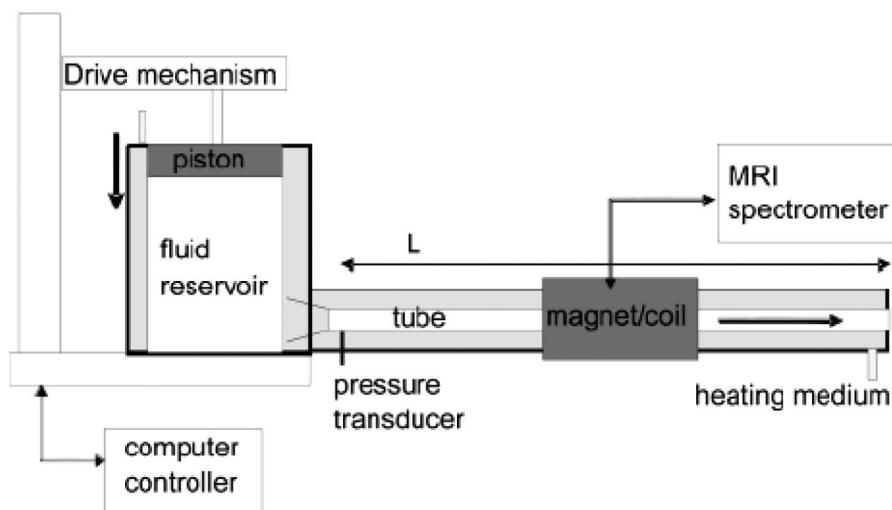


Figure 20 Schematic diagram of the MRI apparatus integrated into tube viscometer (from<sup>[54]</sup>).

effects due to apparent reduction in viscosity and dependence on tube dimensions in measuring fluid properties. In addition, slippage at the tube walls is also another major source of error which can be corrected by using a tube with rough walls.

The magnetic resonance imaging (MRI) has been integrated as part of the tube viscometer apparatus in order to ensure fully developed flow through MR velocity images and evaluate rheological parameters, including the yield stress. The MRI allows to directly image the velocity distribution across the tube and to measure the boundary conditions, so that assumptions of velocity at the wall can be avoided (e.g., slip or stick boundary).<sup>[53]</sup> Figure 20 is a schematic diagram of the MRI-integrated tube viscometer.

Yoon and McCarthy<sup>[55]</sup> investigated the flow behavior of yogurt during pipe flow with MRI and found that the average yield stress and consistency index ( $K$ ) were higher at 25°C (14 Pa and 5.6 Pa s<sup>n</sup>) than at 35°C (10 Pa and 1.1 Pa s<sup>n</sup>); whereas flow behavior indices were the opposite (0.36 and 0.88). They also studied the volumetric flow rates of three commercial processed cheeses during melting with the objective for further mixing and pumping of melted processed cheese.<sup>[56]</sup>

## APPLICATIONS OF YIELD STRESS IN FOODS

### Concentrated Suspensions

Most foods are dispersions of solids in a liquid or one fluid in another. Concentrated suspensions are dispersions of solids suspended in an aqueous medium with concentration varying from 5 to 24% by weight. Yoo et al.<sup>[57]</sup> measured the static and dynamic yield stress of structured and unstructured commercial food suspensions with the vane method at controlled shear rate and shear stress. The yield stress has also been used to evaluate the structural recovery of a disrupted sample to its original consistency.<sup>[38]</sup>

Additionally, the role of particle size and particle concentration on the yield stress of dispersions has received considerable attention. In general, the magnitude of yield stress of food suspensions increases with increasing particle volume fraction, decreasing particle

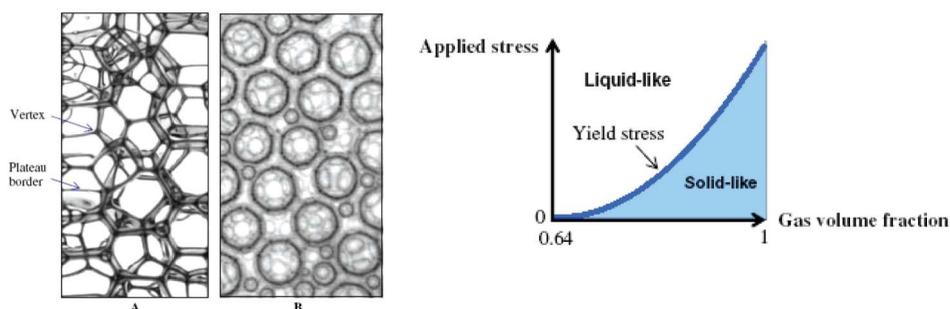
size, and increasing interparticle forces.<sup>[58]</sup> Qiu and Rao<sup>[59]</sup> demonstrated that the yield stress of apple sauces increased with increase in pulp content and with decrease in average particle size. However, Missaire et al.<sup>[10]</sup> showed that differences between structured and unstructured suspensions can cause divergence of results. The yield stress is considered as a measure of interacting particles behavior and the microstructure. Several theoretical models have been attempted to relate yield stress with particle-particle interactions, volume fraction, and particle size.<sup>[58]</sup>

## Foams

Foams are widely used in food applications, either as finished products such as meringues and whipped toppings, or as inclusions during production such as in ice creams and batters for baked goods.<sup>[60]</sup> Foams consist of a colloidal system containing discontinuous air phase in the form of tiny air bubbles dispersed in an aqueous continuous phase.<sup>[61, 62]</sup> In general, foams are classified as either dry or wet, as well as according to their rheological behavior, solid-like or liquid-like. In both cases, the distinction is based on the gas phase volume ( $\phi$ ), as shown in Fig. 21.

At low  $\phi$ 's (below approximately 70%), bubbles maintain their spherical structure, are called "bubbly foams" and behave as a viscous fluid; and if it is high, bubbles are pressed and packed together forming polyhedral foams and take the form of dry foams, thus displaying a solid-like behavior.<sup>[61-65]</sup> Due to their lyophobic characteristics, foams are intrinsically unstable, and as a result, their structure tends to collapse within few seconds to days.<sup>[62,64]</sup> The addition of surfactants, such as proteins, to colloidal systems enhances foam stability. The main food proteins used are whey, egg white, and soy.

The most common methods to evaluate protein-based foams are measurements of overrun and stability. Solution properties such as ionic strength, pH, ion types and concomitants strongly influence foaming functionality and characteristics.<sup>[61,63]</sup> A common practice to examine foam formation is to determine how well it will hold up on a spatula or maintain its structure.<sup>[14, 60]</sup> Thus, observing how well the foam resists flow under the influence of gravity is a subjective measure of yield stress. Objectively, however, the yield stress of foams can be measured with conventional rotational rheometers such as parallel-plate, cup-and-bob, and cone-and-plate, and with capillary flow. Prud'homme and Khan<sup>[66]</sup> obtained good rheological data by adding sandpaper to a parallel-plate rheometer



**Figure 21** (A) Structure of dry; and (B) wet foams (on Left). Schematic diagram of the solid-like and liquid-like behaviors of foams (on right) (from<sup>[64]</sup>).

to prevent wall slip. Similarly, the results from squeezing-flow tests were useful to monitor consistency of foams and to detect gum addition.<sup>[38]</sup>

Pernell et al.<sup>[60]</sup> demonstrated that the vane technique can be successfully employed in measuring the yield stress of protein-based foams made at various concentrations and whipping times. In addition, the point method (controlled rate mode) showed superior results compared to slope method (controlled stress mode). The yield stress values were similar for egg white protein (EWP) foams in both testing modes, but not for WPI foams. It was suggested that the latter create different properties when measured in different ways (i.e., difference in mixer models), while EWP foams were more stable and isotropic. Vane yield stresses for EWP foams were greater than those of WPI foams (100–150 Pa and 55–80 Pa, respectively), despite similar proteins volume fraction, bubble size, and surface tension. Based on this, many investigators had focused on determining factors that influence not only the stability and overrun of protein-based foams, but also their yield stresses.

Overall, yield stress values depend on the effects between the protein absorption at the surface and the protein distribution in the lamellae between air bubbles, rather than just volume fraction or bubble size.<sup>[62]</sup> Additionally, interfacial measurements of surface tension and interfacial dilatational elasticity suggest that variation in the yield stress among protein foams are related to the ability of the protein to form strong and elastic interfaces throughout the surface.<sup>[65]</sup> Luck et al.<sup>[63]</sup> observed that the yield stress of WPI foams is also dependent on pH, salt type, and salt concentration, since these factors alter the foam structure and behavior at the surface.

From the theoretical model of Princen and Kiss,<sup>[67]</sup> the increase in yield stress of foams is proportional to the cubic root of its air phase volume (i.e.,  $\tau_o \propto \phi^{1/3}$ ), as large  $\phi$ 's result in tighter packing of the air bubbles. However, this relation is not entirely true when comparing foams made of different proteins. Although it is intuitive to directly correlate foam stability with yield stress, previous studies have shown that yield stress of foams may be inversely related to their stability. For instance, it is well known that adding sugars (e.g., lactose) to foams increases stability due to an increase in viscosity and decrease in the rate of liquid drainage<sup>[61]</sup>; thus, yield stress and overrun of foams containing lactose decreases.<sup>[63]</sup> Foams of hydrolysate proteins result in highest yield stress values and overrun abilities, though they present poor foaming properties.<sup>[61,63]</sup>

Mleko et al.<sup>[68]</sup> demonstrated that pH-induced refolding of egg albumin produces substantially firmer foam than untreated egg albumin. Foam firmness was evaluated by the static and dynamic yield stress measured with steady shear experiments using a special cross-hatched geometry that prevents slippage. The authors pointed out that their findings can produce more stable and firmer products where egg whites are employed such as in meringue. In addition, pH treatments may improve overall performance and functionalities (e.g., texture) of protein-based foams in foods.

## Concentrated Emulsions

The common measurements of emulsion stability are average particle size of emulsion droplets and evaluation of the thermodynamics of the system, especially at the interface of dispersed and continuous phases. Additionally, viscosity and yield stress are also provide information on how the spherical droplets are packed and interact among each other.<sup>[69]</sup> While the maximum packing of uniform spheres consist of 74% of the space with the remaining 26% of the space is void, at volume concentrations between 60 and

70%, the contact between droplets affects sensible properties that may cause phase inversion (e.g., from O/W emulsion to W/O). Morrison and Ross<sup>[69]</sup> pointed out that at concentrations greater than 70% by volume, emulsion takes the characteristics of polyhedral foams due to the packed structure features.

Moreover, the flow of concentrated emulsions is restricted by the interparticle interactions among the droplets. As the concentration increases ( $\geq 70\%$ ), droplets are tightly packed together and flow only occurs after a large shearing stress is applied to overcome the structure built up. Thus, the increase in both yield stress and apparent viscosity are associated with increasing particle volume fraction, decreasing droplet size, and increasing magnitude of interparticle forces.<sup>[58,69]</sup>

Mayonnaise is an oil-in-water emulsion (vegetable oil in aqueous solutions of mainly egg yolk, sugars and salts) with a volume fraction of approximately 65% in the dispersed phase. The yield stress relates to stability of mayonnaise in low-stress situations such as during storage and transportation. Ma and Barbosa-Cánovas<sup>[70]</sup> reported that the yield stress of mayonnaise increased significantly with increase in added oil and xanthan gum, from 23 to 235 Pa and 55 to 195 Pa, respectively. The added oil promoted a more compact 3-D network formation between the egg protein molecules and absorbed droplets, while added xanthan gum increased the stability of mayonnaise emulsion through the formation of large aggregates. The structural stability of light and regular-fat content mayonnaise products has been compared by measuring their yield stress.<sup>[71]</sup> The light mayonnaise exhibited higher yield stress, which indicated that the regular mayonnaise can be pumped more easily. The light mayonnaise requires an added artificial dispersant to prevent destabilization. Salad dressings are another common O/W emulsions with a minimum 30% volume fraction of the dispersed particles.<sup>[69]</sup> Wendin and Hall<sup>[72]</sup> investigated the effect of fat, thickener, and emulsifier content on salad dressings from creep-recovery tests. Higher yield stress values were obtained for samples containing high fat and thickener contents, and yield stress and viscosity parameters correlated well with sensory panel results.

### Chocolate Coating

The yield stress of foods, such as melted chocolate, directly affects the coating thickness. For instance, if the yield stress is very low, only a small amount of coated layer will remain on the product, which may be undesirable to the consumer. On the other hand, if the material exhibits high yield stress, the coated layer may be too thick, which may be undesirable to the manufacturer. The coating thickness can be measured by studying the material flow on an inclined plane described earlier. Flow occurs when the shear stress exceeds the yield stress, while the fluid remains on the plane if yield stress is much higher than the maximum shear stress.<sup>[15]</sup> As a result, the maximum coating thickness ( $h_{\max}$ ) can be calculated as<sup>[15]</sup>:

$$h_{\max} = \frac{\sigma_o}{g \cdot \rho \cdot \sin \theta}. \quad (15)$$

where,  $\sigma_o$  and  $\rho$  are the yield stress and density of the coating material respectively;  $g$  is the acceleration due to gravity; and  $\theta$  is the angle of the inclined plane. Equation 15 assumes that the coating material does not flow off of the surface. Similarly, another

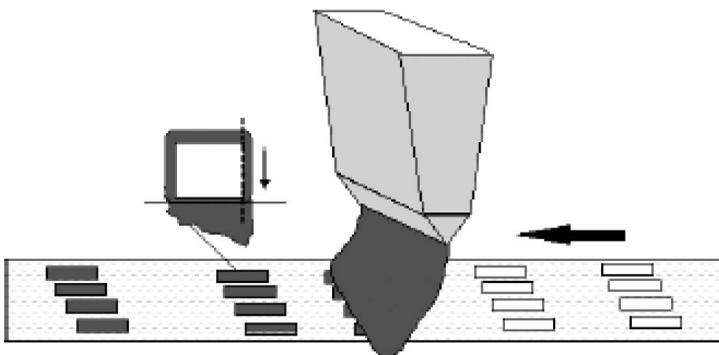
expression for  $h_{\max}$  is given based on post-withdrawal drainage conditions, where  $\theta = 90^\circ$ <sup>[15]</sup>:

$$h_{\max} = \frac{\sigma_o}{g \cdot \rho}. \quad (16)$$

In the chocolate manufacturing and confectionary industry, coating is performed with the enrobing technology, in which the molten chocolate flows in a sheet above a moving belt and the product is coated as it moves through the sheet (Fig. 22). The performance of enrobing depends both on the physics of coating flow and the rheological properties of chocolate.<sup>[54]</sup> As for the latter, in addition to the yield stress, the apparent viscosity is also important.

The International Office of Cocoa, Chocolate, and Confectionary (IOCCC) has adopted the Casson model as a standard to describe the flow behavior of chocolate.<sup>[15]</sup> The IOCCC recommends the use of rotational viscometer with a concentric cylinder probe for flow measurements; several studies have suggested additional recommendations such as measuring shear stresses at specific shear rates (e.g., 2, 5, 10, 20, and 50 s<sup>-1</sup>).<sup>[54]</sup> Recognizing this possibility, Wichchukit et al.<sup>[54]</sup> evaluated the rheological properties of molten chocolate during steady pipe flow using a magnetic resonance imaging (MRI) tube viscometric method, which provides velocity profile and flow behavior during pipeline flow. The yield stress values from the MRI tube viscometry were compared to measurements from rotational viscometer as recommended by the IOCCC and from the vane method. The researchers measured the yield stress values as a function of emulsifier concentration.

The Casson model appeared to fit well the experimental data for all three measurement methods. The MRI images showed that the velocity profile was maximum at the center of the tube, which is expected. The maximum velocity was reached with increasing amount of emulsifiers, whereas both yield stress (15 to 1.9 Pa) and apparent viscosity (14.6 to 6 Pa·s) decreased. Comparing the MRI results to those from rotational viscometer, the latter were lower due to possible wall slip effects, while vane results were in good agreement. Wichchukit et al.<sup>[54]</sup> suggested that the tube viscometer measures static yield stress, as the data correspond to the shear stress to initiate flow. Another aspect of their work was to simulate the average film thickness of milk chocolate during enrobing from



**Figure 22** Schematic of chocolate coating application with enrobing technology (from<sup>[54]</sup>).

an unsteady state mass balance. The numerical solutions were in agreement with experimental results, and as emulsifier level increased from 0 to 0.3%, the predicted thickness decreased by 60%.

### Spreadable Foods

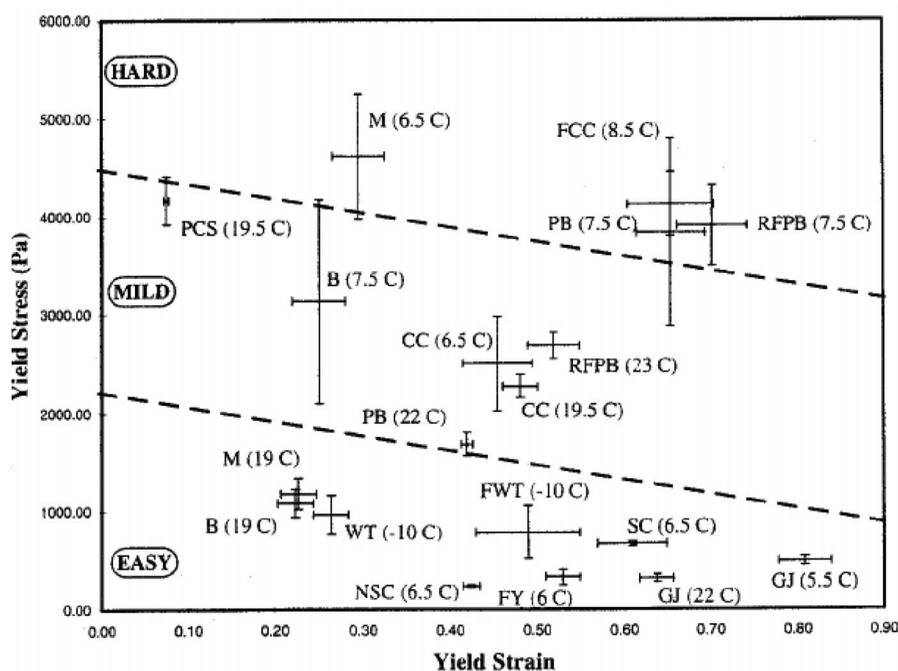
Spreadable foods such as butter, margarine, peanut butter, and cream cheese are elastoplastic or viscoplastic and exhibit yield stress. Early studies by Kokini and Dickie<sup>[73]</sup> on psychophysical mechanisms and fluid mechanics suggested that subjective spreadability of foods could be assessed through the back and forth motion of a knife, and consequently, by estimating the shear stress on the knife surface. This relation may be illustrated as the reciprocal of the maximum shear stress required to create a uniform distribution over a surface<sup>[74]</sup>:

$$\left( \begin{array}{c} \text{subjective} \\ \text{spreadability} \end{array} \right) \approx \frac{1}{\tau_{\max}}. \quad (17)$$

In fact, several studies indicate that yield stress is a better measure of spreadability of semi-solid foods than apparent viscosity.<sup>[33,41,75]</sup> According to Daubert et al.<sup>[74]</sup>, yield stress can be associated with quality and consumer acceptance of spreadable foods, and it provides additional parameter for quality control. A variety of products have been tested and their spreadability values were presented in yield stress-yield strain texture maps (Fig. 23).<sup>[33,74]</sup> Though yield stress is the major contributor to the spreadability of soft foods, the yield strain provides information relative to the extent of deformation and uniformity upon yielding (i.e., flowing). Consequently, while low yield stress indicates good spreadability, the ability of a soft food to resist deformation at low strains can result in poor spread uniformity.<sup>[33,74]</sup>

Peanut butter is a classic viscoplastic food, which consists of a thick concentrated suspension of small non-colloidal peanut particles in peanut oil<sup>[38]</sup>; other vegetable oils and stabilizers may be added into the oil matrix to disperse solid particles. Citerne et al.<sup>[76]</sup> examined the rheological and physical characteristics of two types of peanut butter. One of the products (“100% peanuts”) was an unstabilized suspension with solids and particles in peanut oil, and the second product, referred to as “smooth,” was stabilized with a vegetable oil and contained additives such as salts and sugars. The “apparent yield stress” of both types was obtained by fitting the flow curves, which was measured with parallel plates geometry attached to stress-controlled rheometer, to Casson and Bingham models.

The presence of additives was clearly reflected on the measured yield stress data. The “100% peanut butter” exhibited much smaller yield values, 21.5 and 27 Pa, than the “smooth” type, 363 and 374 Pa, based on Casson and Bingham models, respectively. Similar results were obtained from creep experiments. Transition stress values, denoted by a rapidly increasing viscosity, of 250 Pa and 10 Pa were reported for “smooth” and “100% peanut butter” samples, respectively. Below the transition stress, the sample behaved as viscoelastic solid, and at or above the transition stress as liquid-like material. In addition, Citerne et al.<sup>[76]</sup> observed, from time-dependent investigation with dynamic oscillatory tests in the non-linear region, an elastic plateau at stresses near the apparent yield stress.



**Figure 23** Spreadability map for elastoplastic foods: (M) margarine spread; (PCS) processed cheese spread; (PB) peanut butter; (RFPB) reduced fat peanut butter; (GJ) grape jelly; (CC) cream cheese; (FCC) free cream cheese; (B) touch of butter; (FY) fat free plain yogurt; (SC) sour cream; (NSC) non-fat sour cream; (WT) whipped cream; and (FWT) fat free whipped cream (from<sup>[74]</sup>).

## Cheese

Truong and Daubert<sup>[77]</sup> used vane rheometry as an alternative method for torsion and uniaxial compression tests to evaluate the texture of different cheeses (from hard Cheddar to soft processed cheese). A four-bladed vane was employed to obtain data of stress and deformation from the typical torque-time response at gel fracture. The vane stress was calculated using the relation originally proposed by Dzuy and Boger<sup>[2,32]</sup> shown in Equation 3, and was used to describe the textural properties of cheeses. Meanwhile, the angular deformation was related to the strain characteristic of the material such as in the torsion test.

Comparing the results from the vane and torsion tests, shear stresses obtained from the vane test were lower than those from the torsion test. Similarly, the vane angular deformation at failure were lower than fracture shear strain values in torsion, but both tests showed Mozzarella cheese the most elastic cheese type with highest deformation. Truong and Daubert<sup>[77]</sup> recommended the vane technique for stress and deformation measurements of a wide range of cheeses, especially because it overcomes usual problems with torsion and uniaxial compression tests, which are difficult to perform on soft or sticky and elastic materials, respectively.

Furthermore, the yield stress measured with vane has been used to distinguish ingredients used in cheese. Mleko and Foededing<sup>[78,79]</sup> measured yield stress of rennet casein gels and processed cheese analogs containing various amounts of whey protein isolate (WPI).

The yield stress of the analogs increased with addition of WPI. Additionally, substituting part of the rennet casein in processed cheese analogs by whey protein through heated WPI solutions, resulted in stronger cheese with higher yield values.

### Ice Cream

Ice cream manufacturers constantly modify and develop new products to meet consumers demand such as for reduced-fat ice creams, flavors, and creaminess levels. Briggs et al.<sup>[80]</sup> investigated the viability of testing ice cream with the vane technique and measured the “scoopability” of ice cream based on the yield stress at typical scooping temperatures. They showed that the vane yield stress can be potentially used as a quality control tool, as the yield stress is strongly correlated to body, texture, and scoopability. Yield stress values were also used to identify differences in composition. For example, a less expensive product of brand “B” with lower amount of total solids and higher overrun exhibited higher yield stress than a more expensive product of brand “A.” Furthermore, the correlation between temperature and yield stress was stronger for brand “A” than for brand “B.”

The yield stress was also used as a parameter to study the rheological and structural properties of ice cream mix containing stabilizers locust bean gum (LBG) and guar gum (GG) during sub-zero temperature fluctuations.<sup>[81]</sup> These stabilizers are commonly added to slow down ice crystal growth, especially during recrystallization. The influence of other ingredients (milk solids non-fat (MSNF), sucrose, emulsifiers) was also taken into consideration so that a more realistic system could be mimicked. The yield stress was measured by extrapolation of flow curve data.

Patmore et al.<sup>[81]</sup> indicated that LBG solutions developed viscoelastic weak gels with temperature cycling, especially in the presence of MSNF, as opposed to GG solutions. The formation of weak gel networks around ice crystals reduced recrystallization rates, because the networks formed entraps melted water and prevented it from freezing onto other ice crystals. The addition of MSNF increased the yield stress overall, though did not cause GG to gel. Meanwhile, fat droplets seemed to interfere with the intermolecular interactions in MSNF/LBG emulsions, which were shown by a decrease in the yield stress.

### Yogurts

The yield stress in yogurts is associated to the structural characteristics of yogurt gels. In some types of yogurts, such as fruit-in-the-bottom set types, a considerable firmness is desired to prevent the disruption and breakdown of the structure, whey separation, and better overall texture. Harte et al.<sup>[82]</sup> examined the initial firmness of yogurt based on measurements of yield stress with the vane technique and responses from trained sensory panelists. Yield stress significantly correlated with sensory initial firmness for retail yogurts and laboratory-made yogurts with different amounts of added gum. Yield stress measurements and sensory responses showed that increasing the gum concentration increased initial yogurt firmness.

Yield stress was also used as a parameter to evaluate the characteristics of yogurts as a function of manufacturing process. Harte et al.<sup>[83]</sup> identified differences on the gel firmness for yogurts made from raw milk and from milk subjected to thermal processing (85°C for 30 min), or high hydrostatic pressure process (193 or 676 MPa for 5 or 30 min).

Yogurt gels made from milk treated at higher-pressure for 30 min and thermally processed had similar yield stress values, and both formed firmer gels in contrast to yogurts made from milk at lower-pressure and not thermally processed. In fact, the yield stress values of yoghurts made using milk from the latter two processes were similar. Notably, the effect of variations in milk treatment on the rearrangement of milk proteins and the yogurt gel microstructure was reflected in the yield stress.

In addition to milk processing, the effects of inoculation rate and incubation, not to mention the combination of all three process stages, affect the structure and physical properties of yogurt gels. Lee and Lucey<sup>[84]</sup> examined the effects on the initial gel strength and structural breakdown of intact and stirred yogurts made at different pre-heating temperatures and incubation temperatures. The yogurt gels made from high heating temperature (e.g., 85°C) and low incubation temperatures (e.g., 32°C) resulted in firmer gels with higher apparent viscosity and yield stress values. Low yield stress values of intact yogurt gels made with low milk heating temperature (75°C for 30 min) and high incubation temperature (44°C) indicated that these gels had weaker networks. Moreover, stirred yogurt gels had much less resistance than intact gel, as it would be expected. Thus, higher heating temperature and lower incubation temperature result in higher overall attributes in stirred yogurts.

Similarly, Lee and Lucey<sup>[85]</sup> investigated the effects on the structural network of yogurt gels made at various inoculation rates and incubation temperatures. Yogurt gels made with 0.5% (wt/wt) inoculation rate of starter culture were less stiff than yogurt made with higher inoculation rates (e.g., 2 to 4%). Overall, combinations of intermediate to high inoculation rates along with lower incubation temperatures increase the yield stress of yogurt gels as highly cross-linked and uniformly distributed protein networks are formed.

On the other hand, at lower inoculation rates (e.g., 0.5%), solubilization of colloidal calcium phosphate occurs earlier and at higher pH; thus, the arrangement and interaction of casein micelles in the gel network are changed. Yogurt gels made with lower inoculation rates had lower yield stress values due to weaker interaction between casein particles, causing rearrangement and whey separation more likely to occur.

### Hot Cereals and Starch Dispersions

The yield stress values measured by the vane method was used to characterize the flow behavior of wheat and oat hot breakfast cereals.<sup>[86]</sup> Mimicking the influence of thermal and chemical effects during consumer use, long holding periods of cereals in hot water were determined to increase yield stress, which is caused by the hydration and gelatinization of starch in the cereals. When hot water is added to the mix, the starch granules absorb water and swell. As starch dispersions are gelatinized, the amylose molecules solubilize and leach out of the granules.<sup>[86]</sup> The swollen starch granules become dispersed in a continuous amylose network.<sup>[17]</sup> The vane yield stress values were used as a measure to evaluate the strength of network bonds of cross-linked waxy maize (CWM), tapioca, and Amioca starch dispersions.<sup>[17]</sup> After each of the dispersions has been prepared, sample was let to equilibrate for one hour to recover its structure and reach an equilibrium temperature.

The static yield stress was measured immediately, and the dynamic yield stress after a cyclical flow test had been performed to disrupt the samples' structure. As expected, dynamic yield stress values were lower than the static yield stress values. From the typical torque versus time response, sharpness at the peak stress was more induced with increasing

rotation speed (0.05 versus 2.07 rpm). The authors suggested that at very low shear rates, the vane yield stress would be closer to the “true yield stress.” In addition, at low shear rates (e.g.,  $0.4 \text{ s}^{-1}$ ), the contribution of viscous drag to the total stress was very minimal.

The stress required to break the network structure decreased in the order of cross-linked waxy maize (CWM), followed by tapioca and Amioca. The authors explained using a texture map that the breaking point for CWM dispersion took place at much lower angular deformations, indicating a brittle behavior due to the amylopectin cross-linking present, which was responsible for restricted swelling. Meanwhile, both tapioca and Amioca behaved more rubber-like once they swelled, respectively, very well and moderately well.

## CONCLUSION

Yield stress is a key parameter in various food manufacturing processes and an important tool for product development and quality control. It has been shown that the magnitude of yield stress is strongly correlated to sensory attributes like firmness of yogurt gels, spreadability characteristics, mouthfeel, among many others. Additionally, variation of ingredient sources and texture/structure changes can be detected by yield stress measurement, as yield stress can quantify the level of interacting particles and the microstructure governed by the interparticle network links. Several techniques have been proposed to determine yield stress and many others are being developed and improved continually. Techniques are often classified either as indirect or direct methods, but they may also be categorized as imitative, empirical or fundamental tests. There is no single or “best” method for different foods, and it is not unusual to obtain vastly different yield values obtained for a given product from different techniques. The most appropriate method to measure yield stress depends not only on the availability of the required equipment, but also on the applicability of the measured values.

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