Introduction: Measuring Rheological Properties of Foods

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1 The Evolution of Rheology Through History

Rheology as a discipline has a long history that includes multiple major milestones. Understanding how the understanding of rheology and the precision and accuracy of rheological methods has evolved over time provides a framework of the scope of rheology as a specific discipline. Specifically, this historical background provides the origins of the development of major rheological theories, equations and instrumental devices used in measuring rheological properties. Some introductory level equations are introduced in the following sections of this chapter for the purpose of understanding the necessary fundamental concepts. More details are provided in the subsequent topical chapters, in which the fundamental theories and equations are explained. The milestones mentioned in this section may be considered classic examples for illustrating the methodology and research involved in rheology. Moreover, examples of applications of each milestone to the food industry are provided to allow the reader to interpret major rheological theories in terms of their use in the food industry. A more detailed history of rheology may be found in The Rheology Handbook (Mezger 2014).

Rheology is the science that describes deformation and flow behaviors of materials. Rheology models summarize the physical responses (rheological parameters) under a certain deformation stress or a flow driving force. These parameters (1) reflect processability of crude, or semi-processed or processed food materials, (2) determine physical stability (phase separation rate) of manufactured food products, and (3) describe textural properties of food materials and reveal their sensory attributes. Therefore, rheological parameters and models are essential elements in the food industry for designing processing lines, selecting operational equipment such

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as flow systems and filtration membranes, predicting the physical shelf life of food products, designing formulations for achieving a targeted structure, and predicting sensory attributes.

The term "rheology" was only coined and became a dedicated subject of study in the twentieth century, although rheological research for describing material deformation and flow behaviors was conducted as early as the fifteenth century. One of the first rheological researchers, Leonardo de Vinci, illustrated accurate hydrodynamic and aerodynamic phenomena, such as the flow behavior of air under bird wings and other flying instruments in the 15th and 16th centuries. In 1687, Isaac Newton discovered the relationship between flow resistance and flow velocity; also, he hypothesized the existence of "internal friction of fluids" which is considered a basic law of fluid mechanics and hydrodynamics. In the same year, two brothers, Jacob and Johann Bernoulli defined the theory of elasticity and improved the understanding of hydrodynamic flow and efflux behavior. This theory is still used in the characterization of physical features of fluids and solids. In 1738, Johann Bernoulli's son Daniel Bernoulli described the mathematical relationship between tubular flow behavior and flowing pressures. This relationship is known as the Bernoulli equation, which is used today for computing flow velocity of flowable food materials via flow pressure measurements, including static pressure, pressure due to gravity, and dynamic stagnation pressure. Moreover, hydrodynamic cavitation, which can be calculated using the Bernoulli equation, is considered an innovative process for food sterilization: it may induce cellular inactivation by pressure fluctuations. As the generation of cavities is flow pressure dependent, the Bernoulli equation is the key base knowledge for achieving and controlling the dedicated cavitation effects (Gogate 2011).

In 1839 and 1840, Gotthilf Hagen and Jean Poiseuille designed the first capillary viscometers for characterizing flow behaviors of water, blood, and other low-viscosity fluids. Capillary viscometers are traditional tools for characterizing fluid and semisolid materials; however, this tool has limitations for application in the modern food industry, such as the tubular pinch effect and stick-slip effect. Furthermore, it cannot used for measuring shear-sensitive food materials, as the shear rates in capillary viscometers are quite high (Rao 1977). The mathematical relationships concerning fluid flow behaviors in a capillary tube was consolidated by Gustav Wiedemann and published in 1856 as the Hagen-Poiseuille equation, relating fluid viscosity, volumetric flow rate, pressure drop, length of the tube, and radius of the cross section of the capillary tube. The Hagen-Poiseuille equation plays a critical role in food process engineering; among other uses, it has been used for modeling the permeate flux in the ultrafiltration process of skimmed milk in hollow fibers (Chiang and Cheryan 1986). It is also useful for calculating pressure drop in pipes, and thus sizing pumping equipment.

In 1851, Georges Stokes, the founder of modern concepts of flow dynamics, used "falling-ball" experiments to develop Stokes' Law, a measure of resistance to flow under gravitational forces. In a dispersion system, Stokes' Law models the relationship between buoyancy forces, flowing (creaming or falling) particle diameter, velocity of the flowing particle, and viscosity of the continuous phase. Particle

creaming or sedimentation velocity may be computed using Stokes' Law; therefore, this law is widely used in the food industry for predicting or characterizing the physical stability of emulsion- or suspension-based food products (Maher et al. 2011; Taherian et al. 2008).

The understanding of material viscoelastic behaviors notably increased in the nineteenth century. In 1867–1868, James Maxwell presented "Maxwellian behavior" using viscoelastic liquids. This is considered the first explanation of viscous behavior displayed by solids; the model comprises a spring (elastic component) and dashpot (viscous component) in series, indicating that elastic and viscous behaviors are sequential. Maxwellian behavior in nonlinear oscillatory flow is found in gel-like food materials such as mayonnaise-like emulsions (Hemar and Horne 2000). In 1874, Oskar Meyer presented the Kelvin-Voigt model, one of the first models to summarize the viscoelastic behavior of solids. Generated from oscillatory tests, the model consists of a spring (elastic component) and dashpot (viscous component) in parallel, indicating that these elements react simultaneously to an applied torque or deformation. The Burgers model, developed in 1935, is a derivative model of both the Maxwell model and Kelvin-Voigt model. The Burgers model may be used to characterize the transient viscoelastic properties of solid foods such as cheeses and dairy foams (Karaman et al. 2016; Sharma et al. 2017; Zhao et al. 2015). In 1880, the world's first report on shear-thinning materials was presented by T. Schwedoff, in which he showed that the viscosities of castor oil and gelatin-gel solutions changed as a function of rotational speed of a concentric cylinder system. Shear-thinning flow behavior is one of the key physical features of many food products including ketchup, applesauce, and dairy foods. Accordingly, shear-dependent behavior has been extensively studied and modeled for a wide variety of food systems (Dervisoglu and Kokini 1986; Tárrega et al. 2004). In 1883, the shear-thickening effect was observed by Osborne Reynolds, who also described and discriminated laminar and turbulent flows using the dimensionless Reynolds number. Interestingly, in area of food science and technology, the Reynolds number has been traditionally used to describe flow behaviors of Newtonian and shear-thinning fluids. A specifically modified Reynolds number that properly fit the behavior of shear-thickening food materials was reported only in 2010 and used to model flow behaviors of starch paste and starch–polysaccharide complex paste systems (Ptaszek 2010). The Reynolds number can also be used in heat transfer calculations, as flow regime is a major factor in heat transfer rate. Thus, it is a critical factor in heat exchanger design (Rozzi et al. 2007); Researchers also observed that the Reynolds number affected the fouling rate of whey protein in tubular heat exchangers, likely due to the difference in flow behavior at different flow profiles (Belmar-Beiny et al. 1993).

In 1905, Albert Einstein contributed the Einstein equation in which the viscosity of suspension systems was explained as a function of the viscosity of continuous phase and the volume fraction of the containing particles. This equation opened a door for the exploration of how microstructure of suspension-based food matrices impacts their stability and functionality. A century later, a third-order expansion of the Einstein equation showed better estimation of apparent viscosity of complex

food suspension containing hydrocolloid texturizer and green peas (Chakrabandhu and Singh 2005). The Einstein equation may be also used as tool for predicting viscosity during design of reduced-fat food emulsions (Chung et al. 2013). Around 1913, when the first symposium on "the viscosity of colloids" took place in London, the viscosity of liquid and semisolid materials was considered a material constant which was calculated from measured raw data using the Hagen-Poiseuille equation. Shortly after this symposium, Eugene Bingham defined "viscoplastic liquids" using the Bingham model, and the concept of "yield value" was introduced for discriminating naturally flowable liquids in 1916. Around 1923–1925, A. de Waele and Wolfgang Ostwald reported the famous "power law" model, which expressed the mathematical relationship between shear rate and shear stress for a shear-thinning flow behavior without a yield stress. Currently, this model is one of the most widely used tools for characterizing rheological properties of food materials, for food processing pipeline design, and for food structure design (Sablani and Shayya 2003; Shaker et al. 2000; Verheul et al. 1998; Wu et al. 2013). In 1924, Heinrich Hencky, studying viscoelastic effects, defined Hencky strain, or true strain accounting for deformation of the object, and Hencky strain rate. In viscoelastic food materials such as cheeses, the Hencky strain at the fracture point is thought to be one of the most common instrumental parameters associated with oral textural attributes. Therefore, it was included in a model for predicting textural sensory properties of cheeses, including oral crumbliness, oral firmness, and oral stickiness (Wium et al. 1997). In 1926, Winslow Herschel and Ronald Bulkley modeled flow curves in which "yield point" was considered. In recent decades, the Herschel-Bulkley model is often found in published semisolid food research and is commonly used for designing process parameters (Nindo et al. 2007; Vélez-Ruiz and Barbosa-Cánovas 1998; Gonçalves et al. 2017; Ramaswamy and Basak 1991).

During the last 70 years, due to the advancement of instrumentation and the applications of rheology concepts for solving real-life industrial problems, rheology underwent fast and extensive development. In 1959, N. Casson developed another model to explain flow behavior containing a yield point. Both the Herschel-Bulkley and Casson models are widely used for characterizing the physical nature of semisolid food products; the fit is often compared between the two models (Dervisoglu and Kokini 1986; Gajo et al. 2017; Ramaswamy and Basak 1991). Moreover, an interesting engineering approach showed that the Casson model may be used for computing yield point (yield stress) from experimental data, then the calculated yield stress may be used to generate a better fit to the Herschel-Bulkley model (Tárrega et al. 2004). In 1968, Pierre Carreau introduced the concepts of zero-shear viscosity and infinite-shear viscosity. These concepts are part of a rheological means for describing food structural breakdown (Javanmard et al. 2018), and the zero-shear viscosity is a key rheological parameter that defines the bulk physical nature of a food material. It has also been associated with several sensory attributes including adhesiveness and cohesiveness (Kealy 2006; Nakauma et al. 2011; Selway and Stokes 2013). Additionally, the change of zero-shear viscosity during storage has been used in characterization of shelf life physical stability of yogurt (Lobato-Calleros et al. 2014).

Many scientific instruments have been commercialized, and the measuring methods and systems have been standardized since the 1950s. In 1951, the first rheometer was invented by Karl Weissenberg; the instrument was designed to perform both rotational and oscillatory tests. In 1954, the Haake Company was likely the first commercial manufacturer to launch a rotational viscometer as a product. In 1961, Brookfield Company manufactured a viscometer in which the tests were carried out by pneumatic drive. In 1980s, DIN (Deutsches Institut für Normung, translated as "German Institute for Standardization") standards for the geometry of cylinder systems used in rheometers were published. In the period between 1970s and 1980s, several companies introduced rheometers, including the Rheometrics Company, which became part of TA Instruments in 2002; the Bohlin Company, which became part of Malvern Instruments in 2003; and the Physica Company, which changed its name to Anton Paar in 2004. These three companies are the major interactional manufacturers of rheometers as of the time of publication of this book.

2 Traditional Rheological Tests

Both important terms and concepts used in the food industry and example configurations of some rheology tests are explained in the section. The rheological concepts and tests presented here are all widely used in industry or research applications. This section provides an overview of three major rheological tests: rotational tests, oscillatory tests, and creep relaxation tests. More detailed information on these tests and their use is provided in Chapter "Rheological Testing for Semisolid Foods: Traditional Rheometry".

2.1 *Rheology Basics*

Rheological behaviors are the deformation response to an applied torque or vice versa. In fundamental rheological testing, deformation may be converted to strain and torque may be converted to stress to account for the geometry used for testing. Still, all rheological tests involve measurements of torque and deformation, making these two parameters of critical importance in rheological testing. Understanding a material's response to an applied torque or deformation is the foundation for a complete analysis of its rheological behavior.

Foods are typically classified as fluid, semisolid, and solid. These classifications do not necessarily line up with their rheological classification, which typically is Newtonian (ideal) fluid, viscoelastic fluid, viscoelastic solid, or Hookean (ideal) solid. Hookean solids have a linear relationship between applied torque and subsequent deformation or vice versa. Likewise, Newtonian fluids have a linear relationship between the rate of applied deformation and subsequent deformation. In other words, they have constant viscosity regardless of applied torque. When a torque or

deformation is applied to a Hookean solid, all applied energy is stored in the material and released when the torque or deformation is released, returning the material to its original shape. Newtonian fluids, on the other hand, dissipate all energy imparted to them as heat. Thus, they flow readily under an applied torque or deformation and do not return to their original shape once the torque or deformation is removed.

Few fluid foods display Newtonian behavior; notable examples of such foods include water, honey, and simple sugar syrups. Likewise, even fewer solid foods display Hookean behavior. Typically, only protein gels prepared at high protein concentration (e.g. >10% w/w gelatin gels or >20% w/w whey protein isolate gels) exhibit behavior that can be considered Hookean when tested below strains that induce structural failure. Most foods display viscoelastic behavior. Viscoelastic materials both store energy through their elastic component and dissipate energy through their viscous component. Viscoelastic solids store more energy than they dissipate, while viscoelastic fluids dissipate more energy than they store. Most solid foods are viscoelastic solids with a relatively high proportion of elastic to viscous behavior; most fluid foods are viscoelastic fluids with a relatively high proportion of viscous to elastic behavior. Semisolid foods can be either viscoelastic solids or viscoelastic fluids, depending on their structural features.

One of the main drivers behind rheological testing of foods is to determine their flow behaviors and viscoelastic profiles. Determining these behaviors and how they change under different conditions aids in understanding how foods will behave under industrial processing, storage, and oral processing conditions. For example, a viscoelastic fluid will sag under its own weight and must be stored in rigid packaging, while a viscoelastic solid with little viscous behavior can support its own weight and be stored in more flexible packaging. A more elastic food many be springy or chewy during consumption; a fluid with viscosity that depends on applied torque or deformation may feel different in the mouth when consumed at different rates or while using different oral motions.

In rheological testing of semisolid foods, the food is often placed between two parallel plates (Fig. 1). The upper plate is typically moved while the lower plate is held stationary. During the test, the upper plate receives a shear force over its area. This force results in a subsequent moving velocity of upper plate, causing the edge of the plate to travel a corresponding distance in a given amount of time. Accordingly, shear stress τ (Pa), shear strain γ (unitless), and shear rate $\dot{\gamma}$ (s^{-1}) are defined from the force, deformation, and rate of deformation placed on the sample.

Fig. 1 Two-plate model demonstrating shear associated parameters. (Redrawn and adapted from Mezger (2014))
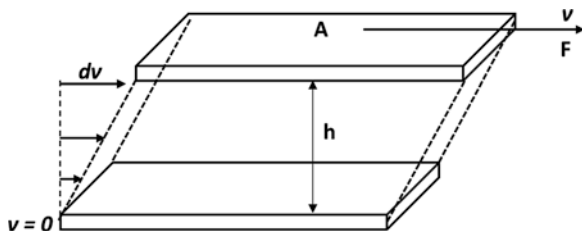
Rheological testing of semisolid foods can be classified into two main categories: rotational and transient. Both categories of tests provide valuable information on food rheological behaviors under industrial or oral processing conditions. Because the rheological behaviors of foods are often complex, multiple types of tests may be needed to fully characterize the rheological behaviors of food products. Typically, the more ingredients a food product contains, the greater the challenge to properly characterize its rheological behaviors.

2.2   Rotational Tests

Rotational tests are often used for plotting flow (viscosity) curves as a function of shear rate or shear stress; thus, these tests are applicable to both fluid and semisolid foods. Rotational tests may be run in two different modes. In controlled strain mode, the correspondent shear stresses are measured at different preset shear rates. In controlled stress mode, shear rate is measured at different preset shear stresses. In both modes, the viscosities (η, Pa.s) are calculated values using Eq. 1:

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{1}$$

Both concentric cylinder and cone-plate geometries can be used in rotational testing. Controlled strain mode is generally used for characterizing flow behavior of materials without a yield point, and it is useful in pipeline flow behavior simulation, as the controlled shear rates may be converted to volumetric flow rate using the pipe radius. This allows the viscosity of material in the pipeline to be calculated at any given flow rate based on the flow curve function. Controlled stress mode is typically used for determining yield point (yield stress) for gelled semisolid foods.

2.2.1   Shear-Dependent Materials

The presence of viscoelastic behavior in a fluid or semisolid material generally manifests as shear-dependent behavior. This means that the relationship between shear rate and the resulting stress is not linear, as it is for a Newtonian fluid. The nonlinearity can manifest as a yield stress, shear thinning, shear thickening, or viscosity plateaus at very low or very high shear rates. Figures 2 and 3 illustrate potential viscosity curves of shear-dependent materials (Levinson et al. 2016; Rao et al. 1997). Most foods with shear-dependent behavior are shear thinning: their viscosity decreases with shear rate. Thus, it is important to know what shear rates are encompassed by the process of interest. For instance, $1–100 \text{ s}^{-1}$ mimics mixing and kneading processes, $10–100 \text{ s}^{-1}$ represents oral processing (chewing and swallowing), and $10–10^4 \text{ s}^{-1}$ represents pipeline flow (Mezger 2014).

Fig. 2 Flow behavior
(viscosity curve) of
different fat-free yogurts.
The decreasing trends of
viscosity as responses of
increase of shear rate
indicate shear-thinning
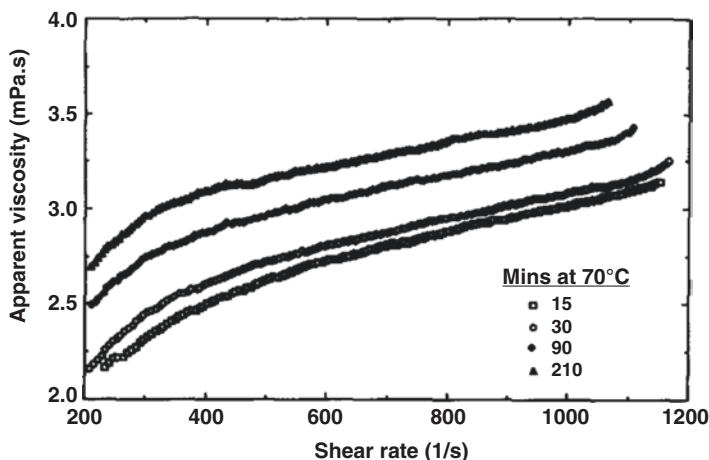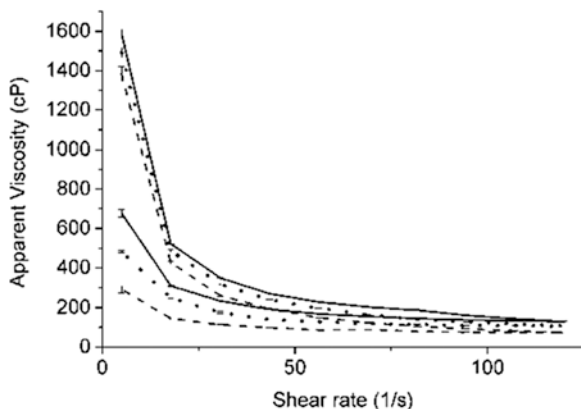flow behavior. (Levinson
et al. 2016)

Fig. 3 Shear-thickening flow behavior of a heat-treated (70 °C) starch dispersion system (2.6%
starch, w/w). (Rao et al. 1997)

It is important to point out that flow behavior for many semisolid food materials
is temperature-dependent. One needs to fix the measuring temperature according to
the specific application need. For example, if the viscosity curve is developed for
simulating pipeline flow at low temperature (e.g. 4 °C), then testing should be
conducted at the same temperature. If the viscosity curve is developed for sensory
prediction, then the test temperature is better set to mouth temperature (37 °C).

2.2.2   Flow Behavior Assessment

As previously mentioned in Sect. 1, multiple flow behavior models have been devel-
oped to account for a wide variety of flow behaviors, including shear-dependent
behaviors, yield stress, zero shear viscosity, and infinite shear viscosity. Shear-
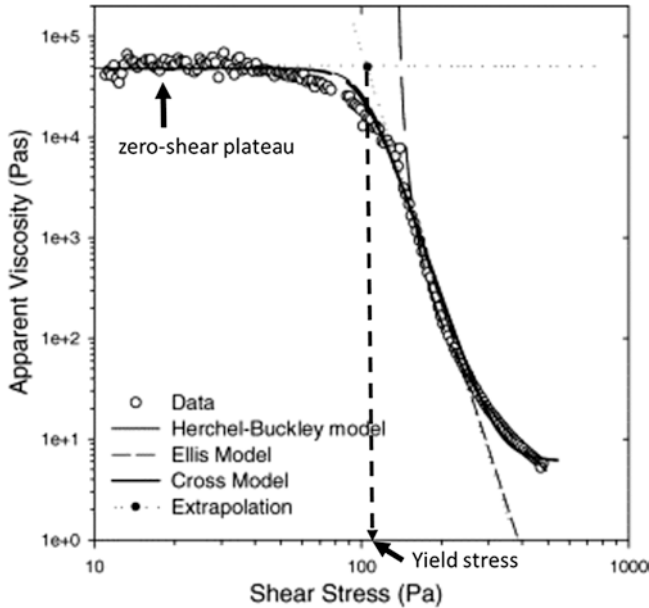
Fig. 4 Viscosity curve of mayonnaise as an example for showing zero-shear viscosity and identified yield stress. The curve is plotted from controlled shear stress sweep method. (Adapted from Stokes and Telford (2004))

dependent behaviors are typically measured by subjecting a material to a series of increasing shear rates and recording the resulting stress. Zero-shear viscosity and yield stress may be measured using a controlled stress sweep method (Fig. 4). Other techniques used for measuring yield behaviors of structured food materials may be found in (Stokes and Telford 2004).

The power law model is one of the most widely used models for food materials and has been used to model the viscosity of stirred yogurt as function of shear rate (Keogh and O'Kennedy 1998). However, the disadvantage of this model is that it does not fit many materials well in the low-shear and high-shear ranges. Because viscosity at low shear rates is important for characterizing the stability of many foods, models that account for zero-shear viscosity are needed for proper modeling of these food systems. Moreover, for a relatively highly viscoelastic gelled food material, e.g. Greek yogurt, a certain amount of force or stress (yield stress) is needed before it starts to flow. Thus, more comprehensive models that express both yield point and flow behavior is needed for better describing a food material. Examples of these models are shown in Chapter "Overview: Semisolid Foods". For additional models and a more detailed explanation of the models presented in Chapter "Overview: Semisolid Foods", the reader is encouraged to review Metzger's The Rheology Handbook (2014).

Certain materials show thixotropic behavior, or thinning due to shearing over time. Thixotropy can be measured instrumentally by conducting a shear rate

Fig. 5 Schematic
illustration of thixotropic
loop and thixotropic area
(shaded)
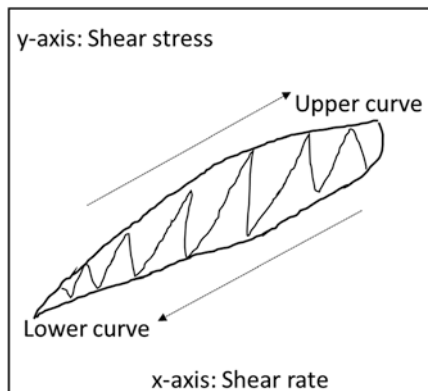
ramp from low to high shear, then high to low shear on the same sample. If
desired, the material can be sheared continuously at the highest shear rate for a
set period of time between the two ramps. Thixotropy manifests as hysteresis
between the two viscosity curves obtained from the shear rate ramps. The thixo-
tropic area may be obtained by subtracting the difference between the areas
under the lower curve from the area under the upper curve (Fig. 5) (Dolz et al.
1995, 2007; Roussel 2006). Thixotropic behavior indicates a structural feature of
food material (Basim 2003). It may govern foaming in some foamed foods
(Rajeev et al. 2008). The impact of thixotropic behavior of foods on the motor
aspect of oropharyngeal swallowing and sensory was also pointed out (Atsuko
et al. 2010; Štern et al. 2008).

2.3   Oscillatory Tests

2.3.1   Basic Concepts of Oscillatory Testing

Oscillatory tests are used to measure the viscoelastic and deformation behavior of
materials. Such methods also known as dynamic mechanical analysis (DMA) are
applicable to liquid, semisolid and solid materials. Parallel plate, cone-plate, and
concentric cylinder geometries may be used to conduct oscillatory tests (Anema
2010; Coviello and Burchard 1992) by oscillating the upper tool at a given fre-
quency (ω) and holding the lower plate or cup stationary. Parallel plate and cone and
plate devices are preferred over concentric cylinder devices, as concentric cylinders
have high inertia and can be difficult to oscillate precisely, particularly at high
frequencies.

Oscillatory tests either import a sinusoidal deformation on a sample and measure
the resulting torque, or impart a sinusoidal torque on a sample and measure the
resulting deformation. Regardless of the input variable, the combination of the
stress and strain waves can be used to determine material viscoelastic behaviors.

Fig. 6 Schematic vector diagram showing G^*, an indicator of viscoelastic behavior (including magnitude and phase angle δ), is a result of integration of G' (magnitude) and G'' (magnitude). (Redrawn based on Mezger (2014))
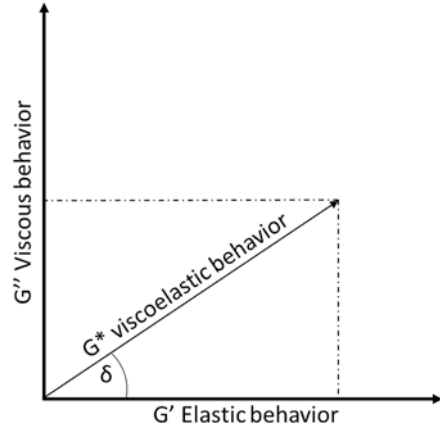

Viscoelastic moduli, including the storage modulus (G', Pa), loss modulus (G'', Pa), and complex modulus (G^*, Pa) can be calculated from stress-strain data; their relationship to each other is shown in Fig. 6 (Mezger 2014). The complex modulus is calculated by dividing the amplitude of the stress wave by the amplitude of the strain wave. Storage and loss moduli are calculated using the phase angle (δ, rad). Storage modulus is an indication of the amount of energy stored in each cycle, while loss modulus is an indication of the energy dissipated in each cycle. In other words, storage modulus represents elastic (solid-like) behavior and loss modulus represents viscous (fluid-like) behavior. The phase angle describes the extent of elastic to viscous behavior; it is usually reported as the tangent of the phase angle (tan δ). When the extent of elastic and viscous behaviors are equal, tan $\delta = 1$. Elastic-dominant materials (viscoelastic solids) have tan $\delta < 1$, while viscous-dominant materials (viscoelastic fluids) have tan $\delta > 1$.

tan δ is an important rheological parameter for characterizing gelled semisolid foods, e.g. dairy gels (Lucey 2001, 2002; Lucey and Singh 1997). For instance, in rennet induced dairy gels, higher tan δ values were associated with higher syneresis (van Vliet et al. 1991). However, while tan δ and G^* values of many foods have been measured (Piska and Štětina 2004; Ring and Stainsby 1985; Svegmark and Hermansson 1991), their statistical correlations with other meaningful characteristics related to their processability, quality, and sensory attributes are poorly understood. Typically, G^* is considered to be an indicator of the overall rigidity of semisolid food materials (Bowland and Foegeding 2001). However, additional work is needed to further understand the significance of G^* and tan δ in terms of food processing ability and sensory characteristics.

Complex viscosity (η^*) is an additional parameter derived from oscillatory tests. η^* is an alternative concept used for describing viscoelastic behavior. Similar to complex modulus, complex viscosity is calculated from a viscous (η') and an elastic (η'') component (Fig. 7) (Mezger 2014). In characterization or quality control of semisolid foods, dairy foods with higher complex viscosity values showed more elastic behavior (solid-like) behavior (Pralle et al. 2000).

Fig. 7 Schematic vector diagram showing η^* as an indicator of viscoelastic behavior is a result of integration of η' (magnitude) and η'' (magnitude). The upper equation is used for calculating η^* if G^* has been already determined from strain sweep data at a given frequency, ω. The lower equation shows how to compute tan δ using η' and η''. (Redrawn from Mezger (2014))
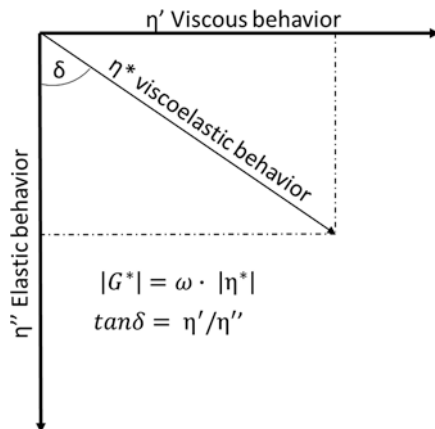
A few notes on oscillatory testing are needed:

All moduli and phase angle values should be determined within the reversible-elastic deformation range, also called the linear viscoelastic region (LVR). This is because the equations for calculating these values, as well as their relationship shown in Fig. 6, only hold in this region. Beyond this region, the material is likely to undergo significant permanent deformation and structural breakdown, which violates the assumption of linear viscoelastic behavior. Furthermore, in standard rheometry, only the amplitude of the input and output waves are used. More recently, mathematical approaches to allow analysis of oscillatory data at high strains and frequencies have allowed rheologists to explore the region of nonlinear viscoelastic behavior. These approaches use the entire output wave to determine additional rheological information about the material of interest. These topics are covered in Chapter "LAOS (Large Amplitude Oscillatory Shear) Applications for Semisolid Foods".

Complex modulus differs from shear modulus (G), as complex modulus is determined from an oscillatory test in which both shear stress and shear strain continuously vary. Therefore, complex modulus is always written with an asterisk to indicate time-dependence. However, both complex modulus and shear modulus can be used to indicate the degree of rigidity of a material and can be used to compare the structural strength of different solid and semisolid foods. Similarly, complex viscosity is measured under transient conditions and is differentiated from viscosity measured using rotational tests with an asterisk.

2.3.2 Amplitude Sweeps

Amplitude sweeps are often used for characterizing the structural nature of food materials. They may be conducted in strain- or stress-controlled mode and are typically conducted at moderate frequencies (0.1–10 rad s^{-1}). Strain (amplitude) sweeps impart an oscillatory strain ramp on the sample and measure the resulting stress response wave; stress (amplitude) sweeps impart an oscillatory stress ramp and
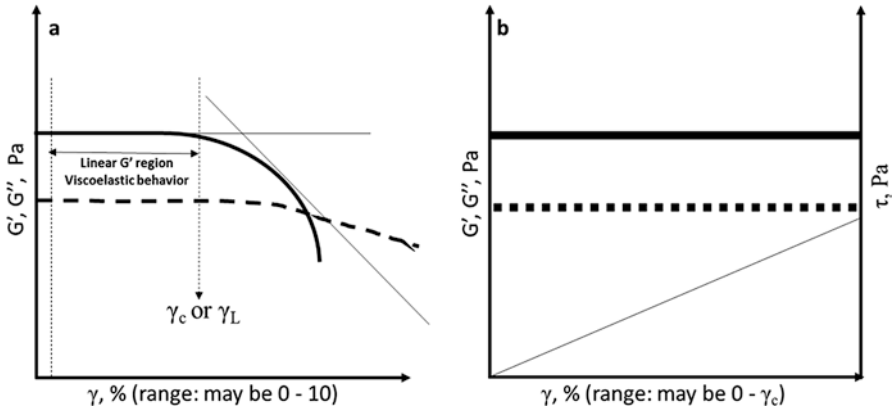
Fig. 8 Schematic illustration of a strain (γ) sweep for a gelled or semisolid food in which elastic behavior dominates viscous behavior. For a, solid line: G'; dashed line: G''; linear viscoelastic region (LVR) is highlighted using a double-headed arrow, critical strain or LVR strain (γ_L) is indicated with a vertical arrow. For b, magnified view of the LVR; bold solid line: linear viscoelastic region of G', dashed line: linear viscoelastic region of G'', diagonal solid line: measured stress (τ) as a function of γ. (illustration is based on example result from Rok et al. (2017))

Fig. 9 Schematic illustration of a stress (τ) sweep for a gelled or semisolid food material. For a, solid line: G'; dashed line: G'', both yield stress (τ_y) and flow stress (τ_f) are indicated with single-headed arrows. (Adapted from Mezger (2014))
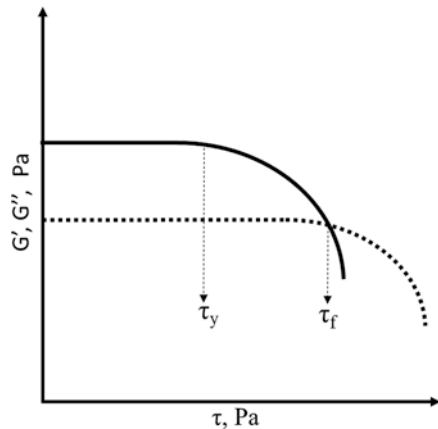
measure the resulting strain response wave. Both tests are performed at constant temperature and frequency. They can be used to measure viscoelastic moduli and phase angle, and thus rigidity of internal structure.

Amplitude sweeps are generally conducted to determine the LVR. To do this, adjacent complex moduli values are compared and the end of the LVR is considered to be when two adjacent values differ by more than 2–10%; the analysis software in most commercial rheometers defaults to 3%. The strain at which the LVR ends is called the critical strain (γ_c or γ_L, Fig. 8a); the stress at which the LVR ends is called the yield point (τ_y, Fig. 9) (Patel et al. 2014). The end of the LVR indicates the greatest stress or strain that can be applied to the material and still maintain the internal

structure before it is disrupted (at the end of the LVR). In the LVR, moduli values are generally assumed to be constant (Fig. 8b), although this is not always true for semisolid foods that do not have a clear yield point. Outside of the LVR, structural disruption results in a decrease in moduli values, and a crossover of storage and loss modulus is possible of the structure is disrupted enough for the material to flow.

2.3.3 Frequency Sweeps

Frequency sweeps impart an oscillating frequency ramp to a sample at constant strain and temperature. This technique evaluates material viscoelastic behaviors at different time scale and can also be used to reveal in-depth insights of structural features of gelled food systems (Tunick 2011). Frequency sweeps can be used to make qualitative comparisons of gel structural type (Fig. 10a) (Tunick 2011) and direct comparisons of gel viscoelastic behaviors as a function of temperature or other treatment variables (Fig. 10b) (Egelandsdal et al. 1986). Soft gels show viscoelastic moduli with a strong dependence on frequency: they may have fluid-like behavior at low frequencies then shift to solid-like behavior at higher frequencies (Stading and Hermansson 1990; Tunick 2011). These gels comprise entangled networks of biopolymer materials, and gelation is a result of polymer–solvent interaction and non-specific conformational entanglement of disordered polymer chains. Food hydrocolloids such as non-gelling starch, locust bean gum, and carboxymethylcellulose can form soft gels (Morris 1986; Stading and Hermansson 1990). Physical gels may show some dependency of frequency, however, a viscoelastic modulus crossover is normally absent and the gels shown viscoelastic solid behavior (Stading and Hermansson 1990). These gels are considered semi-hard gels, and it is believed that non-covalent interactions are involved in their gelation process. Casein micelles can form a physical gel when prepared at acidic pH (pH 4.5–4.6). Hard or strong gels have moduli values that are independent of frequency and show viscoelastic solid behavior (Stading and Hermansson 1990). Covalent crosslinking (e.g. disulfide bonding) is involved in gel structure construction. For example, transglutaminase induces protein gelation networks via disulfide bonding; disulfide bond bridging may also be involved in whey protein gel networks induced by both heat treatment and acidification (Alting et al. 2000; Shimada and Cheftel 1989).

2.4   Creep-Relaxation Test

Creep-relaxation tests are two-part tests that involve stress application and removal (Fig. 11). While a creep test is a transient test rather than a rotational test, it also differs from oscillatory testing in that a constant stress is used. Creep testing may be viewed as a very low-frequency oscillatory test, as it given an indication of the material's tendency to flow over long time periods. In the creep portion of the test, a
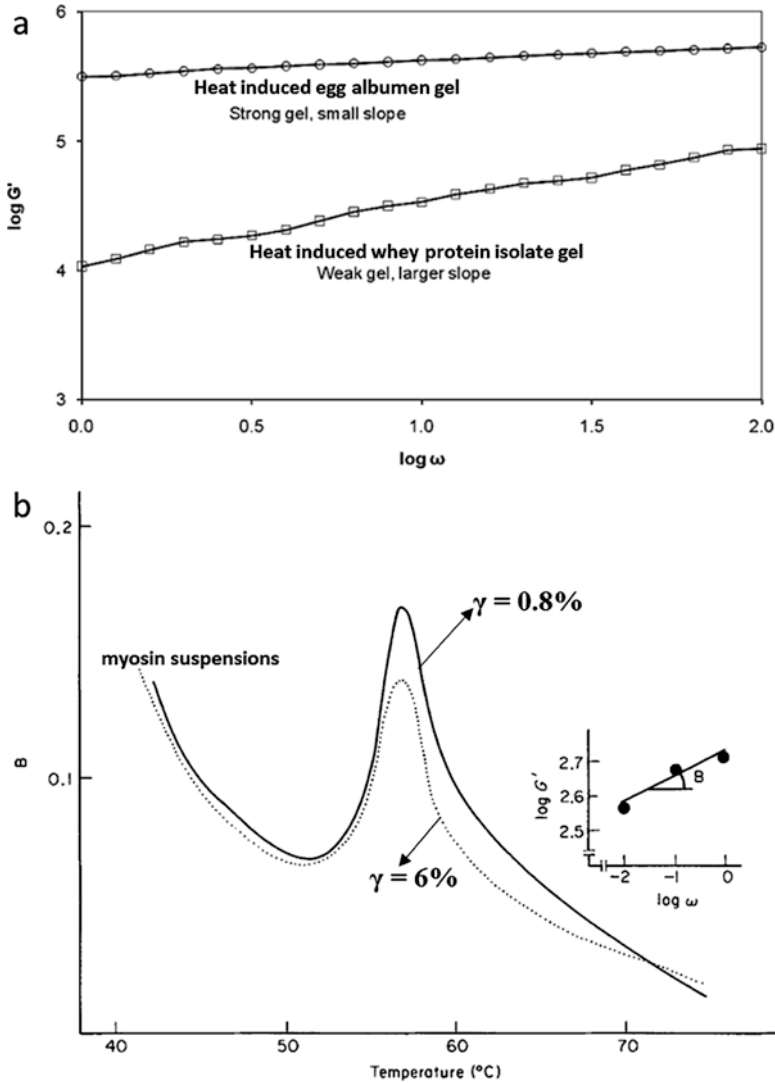
Fig. 10 Application of frequency sweeps for characterizing structure features and structure changes as response of treatments. In (**a**), the upper line shows a relatively small increase in $log(G')$ for an egg albumen gel (20% w/w, induced gelation at 80 °C) with increasing $log(\omega)$; the lower line shows a relatively larger increase of $log(G')$ for a whey protein isolate gel (20% w/w, induced gelation at 80 °C) with increasing $log(\omega)$. The smaller slope of $log(G')$ indicates stronger gel structure (data from Tunick 2011). In (**b**), two different strain amplitudes were used to monitor heat-induced gelation of a myosin suspension (10 mg/mL). The frequency range used was 0.01–1 Hz, B is defined in the inset as the slope of log G′ and log ω. (Data from Egelandsdal et al. 1986)
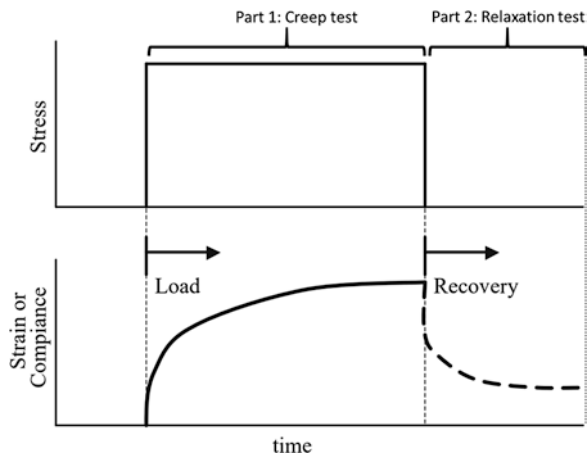
Fig. 11 Schematic illustration of a creep and relaxation test. The upper graph shows the step application of constant stress, holding the stress for a period of time (creep), sudden removal of the stress, and holding for a period of time without stress (recovery). The lower graph shows the measured strain or compliance over the measurement duration. (Redrawn and adapted from Foegeding et al. (2003))

constant shear stress is applied to sample as quickly as possible and the sample is held at that stress for a certain time period. During the relaxation portion, the stress is completely removed and the sample is held at rest for a second time period. Sample deformation (strain) is monitored for the duration of the test. Because creep-recovery testing should be conducted in the LVR, a strain sweep needs to be carried out first to identify the LVR (Foegeding et al. 2003; Foegeding and Drake 2007; Pereira et al. 2001; Tunick 2000). More rigid foods will show a lower strain change during creep testing due to their stronger structure; more elastic foods will show larger recovery during the relaxation portion of the test due to their higher amounts of stored energy. Therefore, creep-relaxation tests can be used to compare structural strength and viscoelastic behaviors of food materials (Tunick 2000). Additionally, creep compliance, or the ratio of strain to stress during creep testing, indicates a material's elastic recoil. This may be important in sensory texture: cheese creep compliance has been significantly correlated with several sensory attributes including hand and chewing firmness, springiness, and adhesiveness (Foegeding et al. 2003).

3   Significance of Rheology in the Food Industry

Some rheological properties of raw food materials and manufactured food products are measured in the food industry as routine quality control parameters. Additional rheological behaviors are profiled or modeled in research and development projects via various rheological techniques. Although the key fundamental knowledge of

rheoloigcal concepts is available in the public domain, it is still a challenge for novice rheologists to properly interpret the significance of individual rheological attributes and rheological measurements in a given application or research publication. A comprehensive review of rheological testing is provided in Chapter "Rheological Testing for Semisolid Foods: Traditional Rheometry". In the following sections, a brief overview of how rheology is used in the food industry is provided, with examples used for illustration of the applications.

3.1   Process Engineering

Many semisolid foods, especially dairy products (e.g. yogurts), show both shear- and time-dependent flow behaviors: their viscosity decreases or increases with flow velocity and over time. Therefore, understanding flow behavior changes in industrial flow systems under different processing conditions is essential for designing manufacturing processes. Factors such as pipeline geometry, pump selection, volumetric flow rate, and heat transfer rate are major processing concerns relevant to rheological parameters including wall shear rate, viscosity and time-dependent flow behavior or pressure-dependent flow behavior. Therefore, modeling of flow behavior is critical to good performance of flow systems. Modeling is one of the engineering approaches for solving industry problems. It has shown advantages over the trial and error approach in food process engineering, as reliable models can eliminate unnecessary trials and predict product flow behaviors in pipelines, therefore reducing research and development costs and processing risks (e.g. improperly sized pumps). Although shear- and time-dependent flow behaviors of semisolid food material in pipelines may be complex, researchers have used multiple approaches for modeling the flow-related parameters of such material. Empirical models (power law-based models) developed via experimental data fitting are widely used for predicting flow behaviors of foods (Butler and O'Donnell 1999; Doublier and Durand 2008). However, these models do not necessarily consider structural or fracture-related factors. A structural approach was applied in investigation of stirred yogurt as a thixotropic fluid; the authors constructed a prediction model which was able to predict friction coefficient in a horizontal rectilinear cylindrical pipe. The model described relationships between pressure drop, an important processing parameter that indicates the suitability of the process for transporting the given fluid, Reynolds number, fracture properties, and shear stress (Schmitt et al. 1998). It should be noted that flow behavior models are much more commonly applied to food process design than structural models, as flow behavior models tend to be simpler to work with and food flow behaviors are relatively easy to measure. Another modeling technique in use for food products is finite element analysis, a diverse and useful numerical tool used to find approximate solutions for complex problems (Hughes 2012; Puri and Anantheswaran 1993; Szabó et al. 1991). For example, it was employed to predict the pressure drop of cultured dairy foods at certain flow rates (Butler and O'Donnell 1999).

Often, it is necessary to predict food rheological behaviors under specialized conditions. Extrusion stretching is normally involved in industrial manufacture of mozzarella cheese, among other products. Accordingly, capillary extrusion analytical techniques have been used to determine flow curves in which different length-to-diameter ratios of the tube were considered (Muliawan and Hatzikiriakos 2008). The modeled flow curves reflected indications of processability of the cheese in realistic situations. Additionally, filament stretching rheometry has provided an opportunity for quantitatively expressing how foods react when stretched under different conditions. For example, the batch filling processability of non-Newtonian foodstuff was found to be a combination of heat transfer and visco-elasto-capillary necking (Tripathi et al. 2000).

In yogurt manufacturing, yogurt milk often blends with hydrocolloids, or thickeners and texturizers, such as starch and other polysaccharides (e.g. pectin). During the pasteurization step of yogurt production, the hydrocolloids completely hydrate, which alters the rheological properties of the yogurt milk. In addition, the non-Newtonian stirred yogurt gel needs to be cooled to storage temperature using a plate heat exchanger after fermentation and smoothing of the yogurt curds. The heat transfer during this cooling process is conducted by both conductive and convective heat transfer. It has been found that the convective heat transfer coefficient during yogurt cooling via plate heat exchanger is related to the Prandtl number, or the ratio of viscous diffusivity to thermal diffusivity, and the design of the plate heat exchanger. The Prandtl number is highly dependent on the dynamic viscosity. Thus, flow behavior does not only govern pipeline flow processes but also influences the efficiency of heat transfer processes for semisolid dairy foods (Afonso et al. 2003).

3.2 Product Stability

Many semisolid foods are oil-in-water (o/w) emulsion or suspension-based systems that are governed by Stokes' Law:

$$v = \frac{2gr^2\left(\rho_2 - \rho_1\right)}{9\eta_1} \tag{2}$$

Here, v is the creaming or sedimentation velocity (m/s), g is the gravitational acceleration (m/s^2), r is the radius of the particle or emulsion droplet (m), ρ_2 is the density of the oil droplet or particle (kg/m^3), ρ_1 is the density of the continuous phase (kg/m^3), and η_1 is the viscosity of the continuous phase (Pa.s). Therefore, a relatively high continuous phase viscosity may result in a lower creaming or sedimentation rate, yielding a relatively more stable emulsion or suspension system. Moreover, viscosity is one of the essential measurable factors for the prediction of phase separation rate of emulsion or suspension systems when Stokes' Law is used. The stability of an emulsion system is a kinetic concept and dependent on multiple factors,

including the physicochemical nature of the emulsifier and oil, droplet size distribution, and the pH and ionic strength of the continuous phase. As we are focusing on the discussion of rheology in this chapter, only the impact of rheological aspects on emulsion and suspension stability is highlighted in this section. More comprehensive reviews of emulsion and suspension stability are available in the literature; interested readers may refer to (Boyd et al. 1972; Dickinson 1993, 2001; McClements 2007; Rousseau 2000; Tadros 2012).

Emulsion droplets can be stabilized with an emulsifier that forms an interfacial film layer on the surfaces of droplets. Stable emulsion-based semisolid foods contain individual droplets that do not coalesce. Rigid interfaces result in robust emulsion droplets and therefore hinder the coalescence phenomenon, making the emulsion system more stable (Edwards and Wasan 1991). Droplet interfaces may present different physical features such as varying degrees of viscoelasticity, and may be quantitatively described by interfacial rheology, the study of flow or deformation behaviors of thin films at interfacial phases (Elmanan et al. 2008; Rousseau 2000). Food emulsions are complexed systems, as they often contain a mixture of different types emulsifiers, e.g. protein and water- or oil-soluble low-molecular weight surfactants. Competitive adsorption of different emulsifiers may occur, resulting in different interfacial compositions with varying interfacial rheological behaviors, which impact stability (Maldonado-Valderrama and Patino 2010). For example, addition of low-molecular weight surfactants to protein-stabilized emulsion system results in protein depletion from the interfacial layer but increased surfactant concentration on the droplet surface (Mackie et al. 2007). Furthermore, increased concentration of surfactant in a stable emulsion system can result in decreased bulk elasticity of the system (Derkatch et al. 2007). This information implies that bulk rheological properties are impacted by alteration of interfacial rheology (Murray 2002). Therefore, by studying the interfacial rheological properties of emulsion droplets, one can better characterize, predict, or even control the phase stability or physical shelf life of emulsion-based food products (Dickinson 2001; Elmanan et al. 2008; Langevin 2000).

The flocculation of food emulsion droplets over time destabilizes the emulsion system. Emulsion flocculation can be induced when van der Waals attraction is prevalent over other repulsive forces, or when Stokes' Law indicates creaming or settling is likely. Droplet flocculation is often characterized by laser diffraction techniques in which the particle size distribution of the population is revealed or by microscopy (Dickinson and Golding 1997; Dickinson et al. 1992). However, these techniques can only show the flocculation phenomenon or the degree of flocculation after it has occurred. Rheological techniques offer an alternative and sensitive means to understand even weak flocculation effects and may be used to indicate flocculation in real time. Shear rheology has been suggested as an effective experimental method for indicating droplet flocculation in moderately concentrated emulsion systems (Dickinson 1998). Rheological flow behavior models and the factors associated with these models have been found to be relevant to flocculation; therefore, rheological techniques and flow behavior models may be used for characterizing emulsion flocculation (Tadros 2004). For instance, a higher yield point and

plastic viscosity in the Bingham model indicates a more flocculated emulsion; however, this is only qualitative characterization (Tadros 2004). Zero shear viscosity and critical shear stress are also able to quantitatively express the degree of flocculation of an emulsion system; an increase of these parameters over storage time may indicate flocculation. These parameters may be gained from a plot of viscosity as a function of shear stress, as well as from a series of constant stress (creep) tests (Tadros 1993, 2004). The method for measuring zero shear viscosity is introduced in this chapter in Sect. 2.2.2 and further discussed in Chapter "Rheological Testing for Semisolid Foods: Traditional Rheometry".

3.3   Food Structure Design & Sensory Prediction

The sensory attributes of semisolid foods are texture-dependent. The specific texture is a result of dedicated arrangements among different food components; such arrangements are expressed as micro- and macrostructural features. Textural attributes and food quality-related properties are structure-dependent (Chung and McClements 2014; Kilcast and Clegg 2002; Pascua et al. 2013; Wilkinson et al. 2000). Therefore, engineering structures is one of the primary tasks in a food product development project to improve product textural attributes. Because these structural features also control food rheological behaviors, semisolid food rheological properties can be related to their textural attributes. In general, both descriptive sensory evaluation and rheological testing are both analytical means for understanding food textural attributes (Joyner 2018), but rheological data collected from instruments is relatively more reproducible and objective. However, sensory evaluation methods are still essential during product development or quality assurance because only sensory data can determine food liking and preference. Nevertheless, rheological measurements can be useful: rheological tests are less time-consuming compared to sensory tests, so using characteristic rheological parameters as benchmarks is a strategy for shortening the research and development process when large number of ingredients or processing parameters need to be screened.

In non-Newtonian food systems, structure, rheological behaviors, and stability are interrelated: a critical stress is able to trigger a coalescence effect among emulsion droplets, and such critical stress is determined by the volume fraction of added thickening/gelling polysaccharide; non-linear creep test, a rheology method, may be used as a fast tool to assess shear induced structural and stability changes (Santos et al. 2015). At the end of the 1960s, Sherman described a concept of texture profile of solid, semisolid, and liquid food materials using a mathematical perspective (Sherman 1969). The theory consists of three categories of food properties. The primary category consists of composition, particle size, and particle size distribution. Rheological properties such as elasticity, viscosity, and adhesion comprise the secondary category, while the tertiary category contains sensory textural attributes. The theory suggested that the tertiary category attributes may be explained by a number of rheological parameters from the secondary category. Many different

sensory prediction models have been developed since that time. The model development is based on three major steps: (1) developing a fundamental understanding of the mechanisms of perception of a textural attribute during mastication; (2) generating data on a broad spectrum of mechanical and physicochemical properties using proper empirical methodology or mathematical models, and (3) determining significant correlations among instrumental and sensory data.

Creaminess is one of the key sensory attributes that determine consumers' acceptability of dairy products (Richardson-Harman et al. 2000; Soukoulis et al. 2010). However, understanding creaminess is challenging due to the lack of a standard definition for creaminess or creaminess measurements (Frøst and Janhøj 2007). Creaminess is a complex sensory attribute, it may be even considered as a combination of other textural sensory attributes, e.g. thickness and smoothness (Kokini 1987; Kokini and Cussler 1983). In fact, creaminess is considered a consumer term by a number of sensory scientists because it is so difficult to describe and quantitatively measure. Based on these considerations, it is not surprising that more than just rheological parameters are needed for constructing reliable prediction models of creaminess perception. Indeed, rheological properties of both the initial undisrupted food and the food after mechanical and enzymatic structural breakdown are necessary for constructing reliable partial least square (PLS) models of creaminess prediction for semisolid foods (Janssen et al. 2007). In the aforementioned study, the authors basically followed Sherman's three-step method for developing sensory prediction models. First, both the initial structural and rheological features and subsequent enzymatic and structural breakdown during oral processing were hypothesized to relate to creaminess perception. Next, rotational and dynamic oscillatory tests were used to characterize the rheological properties of the food at the beginning of structural breakdown caused by mastication, then principal component analysis was applied to consolidate significant correlations. Finally, partial least squares models containing significant factors were constructed for predicting different sensory attributes.

Aside from rheology, soft or "oral" tribology has been highlighted in recent research due to the significance of friction behaviors in food textural perception (Pradal and Stokes 2016; van Aken 2010). Although food rheology and tribology contribute complementary information for understanding food textures, the two sciences are characterized as two different aspects of food behavior (Chen and Stokes 2012); therefore, the detailed application of tribology in food structure design and sensory evaluation will not be discussed here. Additional information on tribology as performed on semisolid foods is presented in Chapter "Semisolid Food Tribology".

3.4 Food Digestion

Rheological behaviors and digestion kinetics are interrelated (A. Mackie et al. 2016; Morell et al. 2014; Prakash et al. 2014). Recently, investigation of the role of food structure in regulating digestion processes in the gastrointestinal tract has become a

topic of interest in both the scientific community and industry. As previously mentioned, interfacial rheology is related to stability of food emulsions; changes to interfacial rheology and interfacial composition among emulsion droplets during and after consumption may promote macrostructural changes of the emulsion system and may have impact on adsorption of digestive enzymes on the surface of emulsion droplets (McClements et al. 2008). Additionally, not only the composition of food but also the microstructure of food system determine its digestion process during and after consumption (Bornhorst and Singh 2014; Singh et al. 2009). Studies have shown that such changes of bulk rheological behaviors can influence protein digestion and amino acid absorption kinetics of the GI tract. For example, a study on dairy protein foods suggested that gelation of a dairy matrix increase stomach retention time, ingestion of liquid matrices is more favorable for muscle protein synthesis (i.e. better protein absorption), and gelled dairy matrices are a more satiating food (Barbé et al. 2013).

Although (bio-)chemistry approaches are often used for understanding food digestion kinetics, modeling stomach geometry and motility of gastric content are complementary means for revealing a comprehensive understanding of food digestion mechanism in the gastrointestinal tract. Computational fluid dynamics is a mathematical means of numerically solving the governing partial differential equations of fluid flow; it is used for expressing sophisticated fluid flow and deformation behaviors as a function of multiple variables, including mass, acceleration, energy, space, and time (Wendt 2008). For example, Ferrua and Singh used computational fluid dynamics to describe digestion in the gastrointestinal tract and revealed that there was an inhomogeneous mixture of gastric contents in digesting high viscous foods rather than a homogenous mixture (Ferrua and Singh 2010). Additionally, modeling of β-glucan digestion showed that rheological attributes such as viscosity indirectly indicated digestion and absorption processes of β-glucan in the small intestine. It was also found that the change of diffusion coefficient of β-glucan was related to digestion processes and that diffusion coefficients decreased somewhat with increased viscosity (Shelat et al. 2011).

4 Summary

Rheology is a useful tool for characterizing the flow and deformation behaviors of food products. Over its long history, rheologists have developed multiple methods of characterizing material rheological behaviors. Rotational tests are typically used to determine flow behaviors, while oscillatory and other transient tests are used to determine viscoelastic behaviors. Rheological behaviors can be modeled through a variety of equations and play a key role in understanding material processing and sensory behaviors. While rheologists have made great strides in understanding how materials react to imparted torque and deformation, much work remains to be completed before a full understanding of material flow, deformation behaviors is achieved. This is especially true for food products, whose complex structures and inhomogene-

ities make rheological characterization difficult. Nevertheless, rheology remains a valuable tool for product and process development in the food industry.

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