Rheology of foods: New techniques, capabilities, and instruments

Peter K.W. Herh, Steven M. Colo, Nick Roye, and Kaj Hedman

Rheology is the branch of science that deals with the flow and deformation of materials. Rheological instrumentation and rheological measurements have become essential tools in the analytical laboratories of food companies for characterizing ingredients and final products, as well as for predicting product performance and consumer acceptance.

The materials under investigation can range from low-viscosity fluids to semisolids and gels to hard, solid-like food products. A knowledge of the rheological and mechanical properties of various food systems is important in the design of flow processes for quality control, in predicting storage and stability measurements, and in understanding and designing texture. Quality attributes such as spreadability and creaminess are extremely important to the acceptance of semisolid food products by consumers. In the case of food materials, texture is a key quality factor. Rheological behavior is associated directly with textural qualities such as mouth feel, taste, and shelf-life stability. As an example, rheological measurements are useful in storage stability predictions of emulsion-based products such as mayonnaise and salad dressings.

Importance of acceptable food rheology

Consumers use subjective tests to determine the perceived quality of a food product. For example, the determination of fruit firmness is based on the deformation resulting from the physical pressure applied by the hands and fingers. The toughness or tenderness of meats is based on the effort required for the teeth to penetrate and chew the flesh tissue. Food companies use instruments in an attempt to objectify these subjective customer perceptions and acceptance criteria. Objective instruments for texture also rely on deformation of the food material. Toughness can be defined as the maximum force required to slice through a sample. Firmness can be defined in terms of the force required to deform a body of material.

The quality of a food product depends strongly on its formulation. Certain ingredients called modifiers have a larger influence on the finished product’s properties than others. Examples of modifiers are stabilizers, emulsifiers, and structural agents. The type of modifier and its desired effect dictate the type of rheological measuring technique used. An antisettling agent should be tested at low shear conditions, simulating settling in a bottle. A sample with an antisettling agent will only be stable for a specific period of time. Therefore, the desired shelf-life stability must be known or determined. Stability is almost exclusively a consumer preference. Most food products change their appearance and texture upon storage. Therefore, an objective, quantitative method for determining their stability is needed. Rheological measurements can be used to predict shelf-life stability once there is historical data on a given product. Comparing a sample with acceptable shelf-life versus an unknown will provide a measure of shelf-life at the production stage, without the need to wait the weeks or months that could be necessary for the actual test sample to settle or phase separate.

A knowledge of the rheological and mechanical properties of various food systems is important in the design of flow processes for quality control, in predicting storage and stability measurements, and in understanding and designing texture.

In food products, small changes in the amount of additive can have a dramatic effect on the final product. The formulation of popular sports drinks can be used as an example. To produce an acceptable lemon-based drink, 1.5% stabilizer/emulsifier is needed in the water/oil interface. At 1.5%, the drink is water undrinkable. In addition, the oil-based product will show a drastic increase in viscosity with time compared to the water-based drink, which is completely stable. A simple rheological test could be the monitoring of viscosity at low shear rates, establish if the correct amount of stabilizer is added.

In principle, every type of consumable food product has some rheological characteristics. Most readily consumable food products contain ingredients that have a major impact on the rheology of the final product. Food processors and raw material manufacturers have been aware of the importance of viscosity for many years. Today, rheological instrumentation is considered a required analytical tool utilized by food scientists on a daily basis. These instruments are Microsoft® Windows® (Redmond, WA)-based, and measurements are made quickly and easily with the use of straightforward, user-friendly software. The operator simply loads the sample into the instrument and selects the appropriate experiment, and the instrument does the rest.

Food products are complex mixtures of different ingredients where individual ingredients are mixed together to produce a finished product. In many cases, the individual ingredients consist of mixtures
of solid as well as fluid components. Most times, they are not homogeneous, and the properties vary throughout the sample. Traditionally, single-point viscosity tests have been performed using empirical techniques. These simple viscosity experiments express the complex rheological response of a sample into a single parameter, and are not adequate in characterizing or providing insight into the quality of food materials. The ingredients used today are numerous and expensive, and, as a result, the cost for controlling these ingredients is high. Detailed knowledge and an objective, reproducible, multipoint measurement capable of decomposing the rheological behavior into individual components is necessary. The STRESSTECH rheometer (ATS RheoSystems, Bordentown, NJ, and RELOGICA Instruments AB, Lund, Sweden) used in this study provides all of the required instrument features and capabilities.

The rheometer is a research-grade analytical instrument capable of measuring viscous, elastic, and viscoelastic properties of liquids, semisolids, and solids (Figure 1). The instrument was developed for use by the serious rheologist, and provides a very broad measurement range, spanning low-viscosity samples such as fruit juice to more viscous products such as creams and salad dressings, semisolids and gets through hard cheeses and solid-state samples. The rheometer incorporates the following features: wide torque, shear stress, temperature, shear rate, and frequency range; true Microsoft Windows-based operational software; patented Differential Pressure Normal Force; patented Sealed Cell measuring system for testing samples above their boiling point; automatic gap setting; remote diagnostics capability via modem; and automatic inertia compensation. In addition, all ATS RheoSystems rheometers are designed on a modular platform allowing easy upgradability. A wide range of accessories satisfy the most demanding applications with ease of operation.

**User-selectable and quantitative, controlled axial normal force sample loading**

Although transient steady shear and periodic dynamic oscillatory experiments provide information on the rheological properties of food products, they do not completely characterize the system. Concerning food samples, or any complex, two-phase system, the rheology is dependent on the sample's deformation history, loading conditions, and the axial normal force applied during a measurement. For example, stress/strain sweeps were performed on a commercial mayonnaise product. The results of a mayonnaise sample run immediately after loading into the rheometer, and a new sample 300 sec after loading into the rheometer, are shown in Figure 2, where shear moduli \((G' \text{ and } G'')\) are plotted as a function of shear stress. The sample run without the 300-sec rest period exhibits lower properties since the internal structure did not have sufficient time to rebuild prior to testing. To dissipate this residual loading history, it was determined that a 300-sec delay time was required after a controlled normal force loading of 1 Newton. The results indicate that both the linear viscoelastic region and the crossover point of \(G' \text{ and } G''\) are affected by the loading condition.

**Sensory evaluation methods for liquid foods**

Systematic analysis of texture is very important for food product development. Texture is a key quality factor in food. One of the most important textural terms obtained is the analysis of thickness. Consumers usually associate changes in thickness with changes in viscous behavior of food materials. To develop predictive correlation between thickness and rheological properties of foods, it is necessary to understand the deformation process in the mouth. The flow properties of liquids can be di-
vided into two main groups: 1) Newtonian, in which a sample's viscosity is independent of applied shear; and 2) non-Newtonian, in which a sample's viscosity is dependent on applied shear. Figure 4 shows these two types of flow properties on pancake syrup. The results indicate the regular syrup is predominantly Newtonian, while the lite syrup shows non-Newtonian flow properties.

Food industries are especially concerned with variations in taste with changes in flow behavior and viscosity. In general, it is known that increases in solution viscosity substantially decrease taste intensity. Also, it has been shown that increases in non-Newtonian flow decrease taste intensity. These rheological properties would allow the systematic development of food products designed for desired texture and taste interactions. Viscosity-taste interactions are predicted by assuming that the rate of diffusion of the tasting agent to the surface of the tongue is the controlling parameter responsible for the intensity of a tasting reaction.

Quantitative normal force measurements
During mixing or agitation, a viscoelastic fluid will climb the impeller shaft in a phenomenon known as the Weissenberg effect. This can be observed in the home during the mixing of cake or chocolate brownie batter.

If a fluid is Newtonian, the viscosity is a constant and equal to the Newtonian viscosity, and the first and second normal stress differences are zero. However, viscoelastic fluids simultaneously exhibit both fluid-like, viscous, and solid-like, elastic, behavior and stress relaxation responses. Manifestations of this behavior, due to a high elastic component, can create difficult problems in processing and engineering design.

Utilizing the patented Differential Pressure Normal Force Sensor, measurements of normal force, first normal stress difference, and normal force coefficient can be made as a function of shear rate as shown in Figure 5 for a commercial honey product. Figures 6 and 7 illustrate steady shear viscosity (n) and first normal stress difference (N1) for lite syrup and chocolate pudding, respectively. Obtaining accurate data for food materials is complicated by various factors such as the presence of a yield stress, time-dependent and shear-dependent behavior, and chemical reactions occurring during processing (e.g., hydration, protein denaturation, and starch gelatinization). This new measurement capability will create significant advances in the utility of normal stress data for the food industry.

Rheological properties of gelling systems above their boiling point
Food manufacturers and processors relying on the functional properties of aqueous gelling agents are well aware that processing conditions such as time, temperature, and amount of shear during the heating can alter the final viscoelastic properties and thickening power of the resulting gelled network, as well as its gelating ability. To date, acquiring fundamental information on the viscoelastic properties of aqueous gels has been difficult due to the experimental requirement of making small amplitude dynamic oscillation measurements on low viscosity aqueous solutions above their boiling point. Although rheological characterization of these systems at elevated temperatures is extremely important to researchers in industry, until now there has not been a viable method available to produce data on reaction kinetics and rheological properties of aqueous solutions during gelatin.

A measuring system designed specifically for the measurement of viscoelastic rheological properties (G′, G″, tan delta) of solutions above their boiling point is the Sealed Cell shown in Figure 8. The patented Sealed Cell measuring system used in conjunction with the STRESSTECH rheometer allows measurements under pressure with full dynamic oscillation and viscometric capability. The cell employs a noncontacting, air-bearing seal rather than standard O-rings. The air-bearing seal is effectively frictionless, and permits dynamic oscillatory differential testing throughout the frequency range of the instrument. Aqueous samples along with solvent-based systems can be measured above their boiling point.

The gelatin behavior of an aqueous system has been studied. First, a dynamic oscillatory temperature scan was performed from 30 to 120 °C at a heating rate of 5 °C/min and a frequency of 0.2 Hz (Figure 9). The results indicate the sample possesses a low viscosity of 70 mPa s at room temperature, and gelatin starts at a temperature around 63 °C, as shown by the increase in viscoelastic properties. The reaction continues as a function of temperature and time, and the end result is a thick, gel-like consistency sample by 120 °C. Of particular interest are the results above 100 °C, where the data indicate that the properties of the gelled system are stable. The data integrity is maintained well above the boiling point of water. No other device can obtain dynamic oscillatory results, especially on low-viscosity samples, above the sample's boiling point due to the mechanical friction limitation of the cell's seals and bearings.

Rheometer system setup
STRESSTECH is a modular research rheometer with a wide range of measuring systems and accessories. Measuring systems are available as concentric cylinders, cone/plate, parallel plate, double concentric cylinders, sealed/pressure cells, and dynamic mechanical analysis (DMA) of rods, bars, fibers, and films. Special measuring systems for low-volume, high-shear rates, and high sensitivity are also offered. The measuring geometries can be made in stainless steel, titanium, polycarbonate, or any user-defined material. The instrument is supplied standard with a patented Differential Pressure Quantitative Normal Force Sensor for reproducible sample loading history, thermal expansion measurements, and quantitative normal stress measurements. The diffusion air bearing has a low inertia with high axial and radial mechanical stiffness.

The rheometer is operated with a separate power supply unit that should be left on continuously. This reduces start-up times and makes it possible for the instrument processor to maintain values as gap and other user-defined settings.

STRESSTECH HR, a high-resolution version of the instrument, allows measurements at microradian displacement and extremely low applied torque.
Temperature control cells are offered that use circulating fluid, Joule-Thomson effect, and cryogenics covering the range from -180 to 500 °C. All measuring geometries are supported, i.e., cone/plate, parallel plate, concentric cylinder, and solids-in-tension and tension. Several high-pressure cells with an upper range of 5800 psig are available.

Electronic unit

The instrument electronics are contained within the mechanical unit and the instrument is built around a dedicated, high-speed 32-bit central processing unit (CPU). This consolidation enhances performance and versatility due to electrical connections on the motherboard bus, rather than through cables to a separate electronics cabinet. In addition, use of valuable bench space is kept to a minimum. The motor control is based on digital rather than analog drive technology. The unit comes with a built-in diagnostic system and quick diagnostic service port for service engineers. Also included is a modem port for remote control operation and fault diagnostics for service. The electronics power supply is designed to operate on a line voltage between 180 and 260 V or 90 and 140 V and an operating frequency between 47 and 63 Hz.

The rheological characterization of foods provides important information for engineers and food scientists to improve and optimize their products and manufacturing processes.

Software package

RheoExplorer 5.0 software (ATS RheoSystems) is based on the Windows operating platform and runs under Windows NT 98/2000. The standard software package is a true multitasking interface with selectable user levels, thus providing many advantages to the food scientist. It is designed to provide flexibility for configuring and using the rheology system. The computer is not dedicated to simply running the instrument and is available for other use when making measurements. The computer can be used for printing previous results, writing a report, or performing measurements with another instrument.

The software enables a normal PC to be used as the interface to allow the user to control the instrument, and then collect and analyze the resulting data. Viscometry, oscillation under stress and strain control, creep and recovery, constant rate, yield stress, fast oscillation, process control, and project (multiplexperiment linking), time-temperature superposition, and spectrum transformation packages allow the sample to be analyzed via different rheological procedures. Powerful data analysis capability allows model fitting, graph and table customization, and cut/paste operation to all other Windows-based software.

The software includes possibilities to link user-designed methods including instrument setup and zero gapping using the Project Software. The dialogue windows have many storable, editable functions for unique testing requirements, and can be reset to default values using default buttons. An example is the Oscillation Frequency Step measuring program, where stresses, delay times, integration periods, and sample sizes could be set individually for all frequencies. Another example is the zooming function, which is presented both in the Viscometry Stress Step and the Oscillation Frequency Step, allowing any number of steps and increments to be selected. The instrument also performs controlled strain and constant shear rate measurements, and has automatic gap adjustments and thermal expansion compensation using the Differential Pressure Normal Force Sensor. The system enhances measurement reproducibility since the sample loading history is reproduced identically each time.

Rheometers for all user levels and applications

DYNALYSER advanced research rheometers (ATS RheoSystems/RELOGICA Instruments AB) (Figure 10) are modular research level rheometer systems designed specifically to address the challenging and diverse testing needs and requirements of the serious rheologist. The instrument’s capability and performance result from a design and development effort focused exclusively on input and recommendations from rheometer users. The rheometer is designed for testing any rheological significant material, including thermoplastics, thermosets, elastomers, semisolids, and/or fluids systems.

VISCOTECH (ATS RheoSystems/RELOGICA Instruments AB) is an entry-level research rheometer system that is fully upgradeable to a STRESS-TECH unit as the user’s needs and requirements dictate. DSR QC is a dynamic shear rheometer (DSR) designed specifically for routine viscoelastic measurements in the QC laboratory.

All the rheometers described here are produced according to ISO 9001 and are tested to operate according to the electromagnetic compatibility rules within the European Community. The instruments are tested to be labeled with the CE-mark.

Conclusion

This article reviewed the important rheological characteristics of several different food products, and results generated with a STRESSTECH rheometer were presented. In addition, a detailed interpretation of the applications and the rheological response with the physical/chemical properties of different semisolids of food products were detailed. The rheological characterization of foods provides important information for engineers and food scientists to improve and optimize their products and manufacturing processes. Today, most formulators count on rheological measurements to develop customer-favored products with a competitive edge in the marketplace. A reliable research-level rheometer and a thorough understanding of food rheology is now a necessity for food companies.