A system for rheological measurements on samples above their boiling point

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

What determines the marketability of a food product? Most food manufacturers agree the success or failure of a product is dependent on a delicate balance of taste, sensory perception, processibility, and long-term stability. Since there is a strong interaction between these parameters, an objective, repeatable method for correlating the final product performance to processing and formulation is required. Of these controlling parameters, all but taste are directly quantified by rheology.

The air-bearing seal is effectively frictionless and permits dynamic oscillatory testing throughout the frequency range of the instrument.

Rheology is the study of flow and deformation of materials. Rheometry is a powerful and straightforward measuring technique that until recently has not seen widespread acceptance in the food industry due to instrument limitations for measuring samples without loss of the continuous phase. Food quality properties such as mouth-reel and shelf life are directly related to how a food product flows and, thus, its rheological characteristics. Rheology can provide a direct, objective quantification of these parameters for product development and quality control.

Figure 1 *Sealed Cell.*

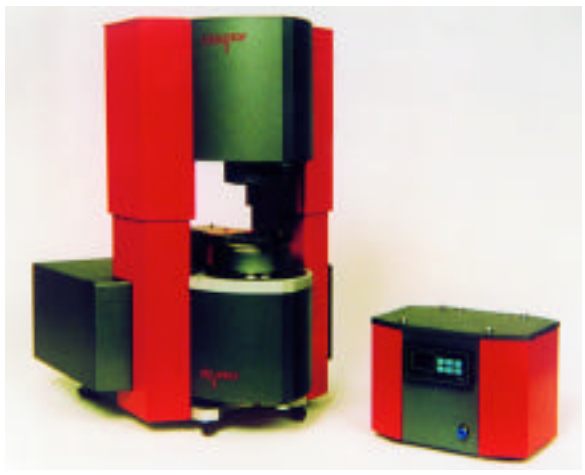

Figure 2 *STRESSTECH Rheometer.*

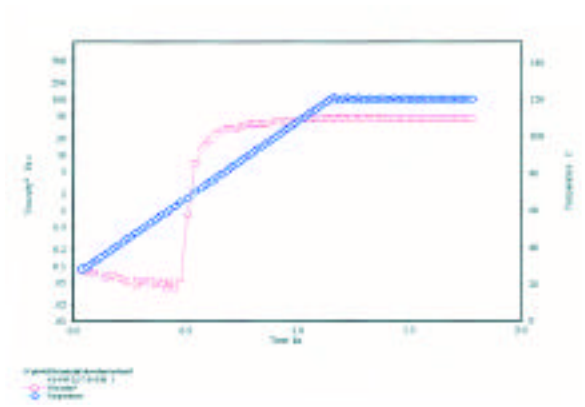

Figure 3 *Gelation behavior of an aqueous system at 120 °C with Sealed Cell.*

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 heating can alter the final viscoelastic properties and thickening power of the resulting gelled network, as well as its gelling 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 industries, there has not been a viable method available to produce data on reaction kinetics and rheological properties of aqueous solutions during gelation.

The Sealed Cell (**REOLOGICA Instruments Inc.,** Bordentown, NJ) was designed to be application specific for the measurement of viscoelastic rheological properties (G′, G″, Tan Delta) of solutions above their boiling point or without loss of any volatile components (*Figure 1*).

The patented measuring system, used in conjunction with the STRESSTECH Rheometer (**REOLOGICA Instruments, Inc.**), allows measurements under pressure with full dynamic oscillation and viscometric capability (*Figure 2*). The cell employs a noncontacting, air-bearing seal rather than standard O-rings. The air-bearing seal is effectively frictionless and permits dynamic oscillatory testing throughout the frequency range of the instrument. Aqueous samples, along with solvent-based systems, can be measured above their boiling point.

The gelation 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 3*). The results indicate that the sample possesses a low viscosity of 70 mPa sec at room temperature and gelation 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, gellike 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. The device obtains dynamic oscillatory results on low-viscosity samples above the sample's boiling point because there is effectively no mechanical friction limitation of the cell's seals and bearings.

ATS RheoSystems (Bordentown, NJ) and **REOLOGICA Instruments, Inc.** offer research rheometers, technical consulting, method development, applications training, contract testing, and technical support and service for the characterization of food products. The instruments are designed for use in research, product development, and quality control with operation through a user-friendly Microsoft® Windows™ (Redmond, WA) software interface. **ATS RheoSystems** assists the scientist by developing the required test protocols and provides data interpretation. The operator simply loads the sample and the rheometer does the rest.

*Mr. Colo is President and Mr. Herh is Applications Manager, **ATS RheoSystems,** 52 Georgetown Rd., Bordentown, NJ 08505, U.S.A.; tel.: 609-298-2522; fax: 609-298-2795; e-mail: info@atsrheosystems.com; Web site: www.atsrheosystems.com. Mr. Roye is Western Region Manager, **REOLOGICA Instruments, Inc.,** North Hollywood, CA, U.S.A.; tel./fax: 818-753-2960; e-mail: info@reologicainstruments.com; Web site: www.reologicainstruments.com.*