

# A new quality and process control technology for real-time testing of viscoelastic changes in dairy and food products

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## ABSTRACT

Measuring the viscoelastic properties of soft food materials with precision for industrial QC and production process control is a continuing challenge. Research laboratory equipments are typically not adapted for industrial use and are costly because of required capital investment and high operational costs (only highly skilled professionals can efficiently operate rheometers). On the other hand, texturometers that are widely used in the industry lack precision and do not allow controlling the production process.

Rheolution Inc. has developed new, simple to use and cost-effective instrument series, ElastoSens™ and CoaguSens™, for real-time quality control and production process control of food products and ingredients. The technological principle of these instruments is purely mechanical and its day-to-day use is much simpler than the use of traditional instruments, such as rheometers. The capabilities of ElastoSens™ will be presented through the results of viscoelastic testing of gelation and melting processes of various ingredients and food products. CoaguSens™, the processing hall version of the technology, was used in cheese production plants for at-line mechanical monitoring of milk coagulation processes and cutting time determination on the basis of curd elasticity. The results showed reduced variability (-45%) in yield after adopting CoaguSens™ as part of cheesemaking process. This allows refining the process and leads to considerable savings upon improved yield and quality. Rapid quantitative measurement of curd firmness facilitates further automation of the cutting step.

**Keywords:** viscoelasticity, quality control, real-time process monitoring, gelation, coagulation, soft food products

## 1 THE CHALLENGE: MEASURING THE ELASTICITY OF SOFT MATERIALS

Viscoelasticity testing during and after the structure formation or structure loss allows elucidating structure-texture property relationships of food and dairy ingredients and monitoring and controlling processes which involve changes in mechanical properties. Examples of such processes include coagulation, gelation, melting, or degradation of products and formulations, such as dairy products, protein- and polysaccharide-based gels, and food emulsions. Currently, it is still a challenge to monitor the mechanical properties of food products in production environments to control quality and processes.

Because of the continuous need to control and modify food ingredients to meet specific textural requirements, or to control industrial processes such as coagulation of milk, Rheolution Inc. has developed a new analytical technology to measure elasticity of food materials both in laboratory and at-line (in plant). The aim of this simple-to-use technology is to measure in real-time the elasticity of soft materials during and after the structure formation without perturbing the process, without destroying the sample and in a cost effective way.

### 1.1. ElastoSens™: Lab QC applications



Most available non-destructive instruments are based on optical methods, focusing on molecular level characteristics (at nanometer-range distances), and few techniques with the exception of certain rheological methods (such as small-amplitude oscillatory shear, SAOS) allow monitoring larger-scale material properties, such as viscoelasticity. However, following the same sample over long periods of time is seldom possible without repeated sample loading and altering the sample properties.

For this purpose, Rheolution Inc. has developed ElastoSens™ X3, the first lab mechanical testing instrument allowing:

- To characterize in real time and non-destructively the viscoelasticity of food ingredients and formulations.
- To measure the mechanical properties without contact and without perturbing the structure formation or loss.
- To use removable and sterilizable sample holders to follow the evolution of the same sample over long periods of time.
- To measure the mechanical impact of physical stimuli such as temperature and pH on food products.

## 1.2. CoaguSens™: Automatic & at-line process control of milk coagulation in the cheese industry



The technology has also been developed as a processing hall-adapted version, **CoaguSens™**, which is used in cheese plants for at-line monitoring of the coagulation of milk during the cheesemaking process. In cheese production, milk gel is usually cut at predetermined time or according to cheesemaker's subjective evaluation of gel texture and appearance in order to separate whey from the cheese curd. These can lead to considerable variation in yield, because many factors affecting the gel strength do not remain constant.

There is a pressing need for objective and quantitative real-time milk gelation and cutting time determination that help refine the cheesemaking process, improve process automation, reduce the variations, and maximize yield. In cheese production, CoaguSens™ allows:

- Measuring milk gel elasticity during coagulation.
- At-line monitoring of milk gel formation in real time.
- Precise and automatic monitoring of cutting time.

## 2 THE TECHNOLOGY

Contactless and non-destructive ElastoSens™/CoaguSens™ technology measures in real time the viscoelastic properties of gels as a function of time and temperature or other stimuli. Both heating and cooling ramps can be applied to the sample. The patented technological principle behind this instrument is purely mechanical. The sample is contained in a cylindrical disposable and detachable sample cup having a flexible bottom. Small and gentle vibration of a few micrometers is applied to the sample and the response is measured using a laser probe without contact (Fig. 1).

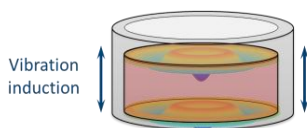


Fig. 1 Contactless and non-destructive viscoelasticity measurements by ElastoSens™ and CoaguSens™ using a few micrometer-amplitude vibrations.

The material response is processed to obtain the viscoelastic properties (i.e., shear storage and loss moduli). This process is repeated in desired intervals to characterize the mechanical evolution of the studied material.

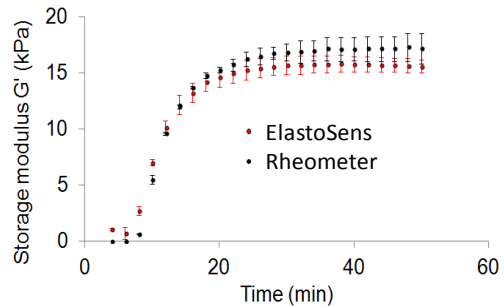


Fig. 2 Comparison of ElastoSens™ X3 with a rotational rheometer (n = 3).

The technology was validated by a comparison with a commercial rotational rheometer that provides identical viscoelastic information. Fig. 2 shows the elasticity results on the same sample (1.4 % w/w solution of cell culture-grade agar), measured by ElastoSens™ X3 and a rotational rheometer ( $f = 1$  Hz, strain = 0.1 %).

## 3 VISCOELASTICITY OF FOOD INGREDIENTS

### 3.1 Gelation at constant temperature

Hydrocolloids are polymers that form viscous dispersions or gels in water. In food industry, they are used as thickeners, gelling agents, emulsifiers, stabilizers and coating ingredients, owing to their ability to modify the rheology of food systems. Common hydrocolloids include agar, alginate, carrageenan, various cellulose derivatives, chitosan, gums (gum arabic, guar, xanthan), pectin, starches and gelatin. The gelation of hydrocolloids can occur via ionic interactions, cold-setting or heat-setting.

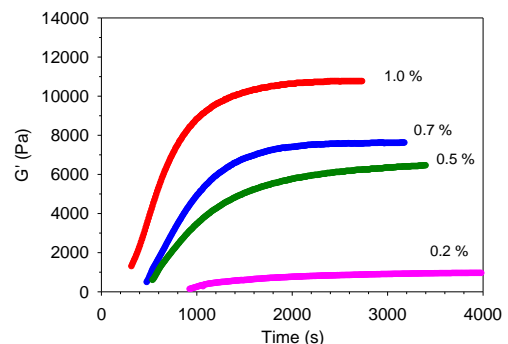


Fig. 3 Concentration dependence of agar gelation kinetics at 25 °C, as measured by ElastoSens™ X3 (n = 3).

Agar is a cold-setting gelling agent that can be used for both hot and cold foods owing to its high dissolution temperature. ElastoSens™ X3 was used for non-destructive monitoring of food-grade agar gelation upon cooling the solution to room temperature without perturbing the gelation process. As the polysaccharide chains of agar are

prone to spontaneous or acid-induced degradation, testing the batch-to-batch variation of gelling properties or the stability of formulations is an important part of quality control and R&D processes.

The gelation of agar was monitored using ElastoSens™ X3 after injecting warm solution (40 °C) in a sample cup at 25 °C. The pH of the solution was 7.4. At low concentrations (Fig. 3), the probability of chain association is lower and thus, the gelation occurs slowly and the gels are weak.

### 3.2 Temperature-induced changes

As described above, thermal gelation of hydrocolloids can occur either via heating or cooling. In addition to structure formation, structure loss is important for textural attributes, usable temperature range, and storage stability. For example, meltability and corresponding elasticity of cheese is important for both processing and use in cooking, and depends not only on the type and composition of cheese but also on the milk processing conditions. Fig. 4 shows an example of the evolution of viscoelastic properties of processed cheese upon heating, as monitored using ElastoSens™ X3. The instrument can be used for testing the quality of formulations or the effect of composition on the viscoelastic properties and thermal transitions without perturbing the processes.

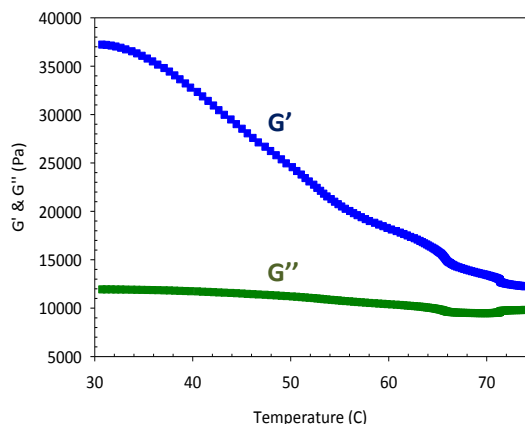


Fig. 4 Gradual melting of processed cheese upon heating, as monitored using ElastoSens™ X3.

## 4 REAL-TIME MONITORING OF MILK COAGULATION: IN-PLANT STUDY

### 4.1 On CoaguSens™

Cutting time selection of milk gels greatly affects the yield, moisture and quality of cheese. Cutting the gel too soon when the curd is not firm enough leads to lower cheese yield through increased curd fines and whey fat loss, whereas delayed cutting results in higher curd moisture content due to reduced collapse of the gel. This may further alter the ripening process and final product quality. Optimizing the process and reducing the variability in cheese yield converts into interesting savings, as the milk price makes most of the cheese production costs.

CoaguSens™ instrument has been developed for precise quantitative monitoring of the coagulation and gelation processes at food processing hall conditions. The instrument comes with CoaguTouch™, a modular, user-friendly and connected touchscreen-based user interface designed to configure the instrument. CoaguTouch™ provides user-oriented tools and functions to manage, analyse, display, store and transfer data and may be used in two different operating modes: 1) Process calibration mode or 2) Process control mode (communication with PLCs).

An example of how CoaguSens™ monitors the milk coagulation is shown in Fig. 5. In this case, the coagulation kinetics and milk gel elasticity depended strongly on rennet (90 % chymosin solution) concentration.

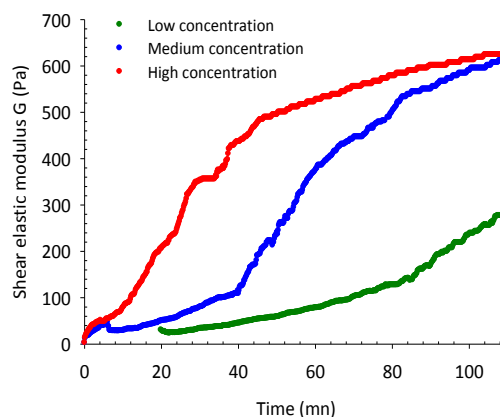


Fig. 5 Gelation of milk at 41 °C after adding rennet solution at different concentrations (0.05, 0.42 and 3.33 % v/v).

### 4.2 In-plant study

The study conducted in a medium-sized cheese plant was divided into two phases to determine the impact of using CoaguSens™ on cheese yield: (I) Database development, and (II) Cutting time control.

**Phase I:** cutting time was determined according to the cheesemakers' subjective evaluation of milk gel texture and appearance while simultaneously monitoring the coagulation process and the evolution of milk gel elasticity (curd firmness) at-line by CoaguSens™. The mean values and variability of milk gel elasticity and cutting time were analyzed.

**Phase II:** the milk gel formation was followed by CoaguSens™, as above, but now the cutting step was manually started when the gel elasticity reached the mean elasticity obtained in Phase I.

Seasonal variation in milk composition was observed between the two phases. In short, milk in phase II was less rich than in phase I. Consequently, the yield was expected to decrease in phase II.

### 4.3 Results

In Phase II, the cutting of curd was started when the elasticity reached the mean value measured in Phase I. This led to greater variability (+ 51 %) in cutting times due to

batch-to-batch variation of coagulation kinetics, but reduced the variability in cutting elasticity by -51 % (Fig. 6).

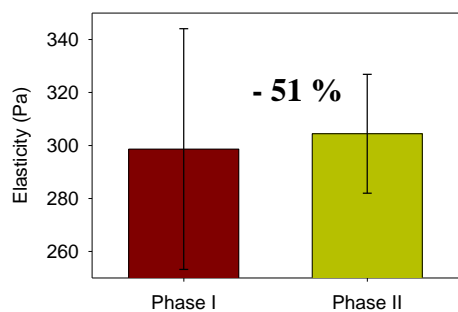


Fig. 6 Mean values and the variability of milk gel elasticity at cutting in Phase I (red) and Phase II (green), without and with CoaguSens™, respectively.

Importantly, reduced variability in curring elasticity was reflected in cheese yield: the variability in actual yield reduced from  $\pm 0.47\%$  to  $\pm 0.25\%$ , that is, by 45 % (Fig. 7). The mean yield in Phase II remained stable compared to Phase I, because the mean cutting elasticity was identical in both phases. The cutting time monitoring by CoaguSens™ prevented the decrease in yield expected due to the seasonal change in milk composition.

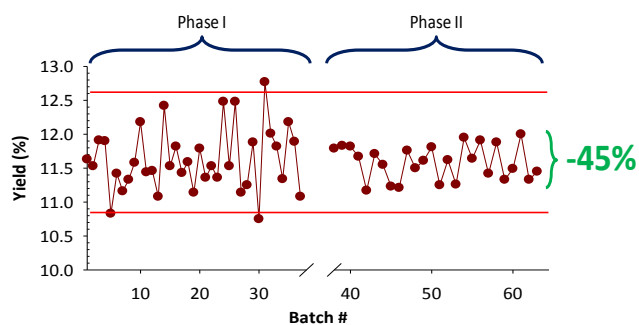


Fig. 7 Cheese yield and its variability in phases I (without CoaguSens™) and II (with CoaguSens™).

CoaguSens™ has a proven capacity to control and help minimizing the variability of the yield of industrial cheese making process. High variability in production process can lead to increase raw material and operating costs, lower plant capacity, and product quality issues. In cheese industry, reduced variability allows improving the manufacturing process and optimizing the yield within the confinements of product regulations (composition, humidity). This has important economic and environmental benefits.

More importantly, CoaguSens™ may be used by manufacturers as a routine process control instrument to set the best cutting elasticity that ensures high yield and optimal moisture content. Using a simple calculation based on milk price in the US and Europe, 0.2 % increase in cheddar cheese yield when using CoaguSens™ in a plant processing 600 tons of milk daily would lead to nearly 2 M USD annual savings. In countries such as Canada or Switzerland, where milk prices are high, the annual savings are even higher.

## 5 CONCLUSIONS

Among the most interesting applications of the new ElastoSens™/CoaguSens™ technology is non-destructive real-time measurement of mechanical evolution of various food ingredients and formulations during and after the structure formation or structure loss without perturbing the process. Measurements are conducted without contact and the samples remain sterile. This affordable and simple to use technology has an innovative feature of removable and sterilizable sample holder that can be stored in a controlled environment to study the long-term stability and viscoelastic changes in different conditions. The measuring accessory with three parallel sample holders can greatly improve the productivity of routine testing and the statistical significance of measurements.

The advantages in QC and production process control are:

- Real-time control of product quality.
- Fast correction of the recipe to reach optimal quality.
- Adjusting process conditions such as formulation and temperature.
- Real-time testing of the impact of additives.
- Stopping gel formation in time.
- Avoiding technical accidents and losses in production.
- Standardizing the product quality.

All these capabilities are made possible by the new ElastoSens™ technology designed to overcome challenges in food and dairy industry QC and process control.

The industrial processing hall version of the technology, CoaguSens™, allows at-line testing of products and controlling production processes through rapid individual tests or continuous monitoring. In cheese manufacturing plants, the data can be used for automated control of processes such as cutting of milk gels at desired curd firmness. Reduced variability in yield and the possibility for process optimization convert into interesting savings.