Mechanical Monitoring of Hydrocolloids for Food Industry using ElastoSens™

How to measure viscoelasticity evolution of rheology modifiers and formulations?

ElastoSens™: the first contactless mechanical testing instrument

For non-destructive monitoring of short- and long-term viscoelastic changes

Industrial Production | Food Chemistry | Process Control

Hydrocolloids, gels, food formulations, food and dairy rheology, non-destructive & contactless testing

BACKGROUND

Hydrocolloids (hydrophilic colloids or gums) are polymers that form viscous dispersions or gels in water. Common hydrocolloids include agar, alginate, carrageenan, various cellulose derivatives, chitosan, gums (gum arabic, guar, xanthan), pectin, starches and gelatin from different sources. In food industry, they are used as thickeners, gelling agents, emulsifiers, stabilizers and coating ingredients. They are widely used as additives because of their ability to modify the rheology of food systems. This primarily means tuning the flow behavior (viscosity) and mechanical solid properties (texture) of foods, which further influence their sensory characteristics.

Examples of food formulations where hydrocolloids are used to achieve desired viscosity and mouth feel include soups, sauces, dressings and toppings. They are also widely used in the texture modification of ice creams, jams, jellies, gel-like desserts, cakes, candies, etc. As the global sales of gluten-free and other specialty products have risen sharply in recent years, hydrocolloids are particularly interesting owing to their ability to mimic the viscoelastic properties of gluten or to replace fat or eggs in food formulations.

Because of continuous need to control and modify the ingredients for high-quality food products, much of the recent research has been dedicated to non-destructive analytical techniques to elucidate structure-property relationships in foods. The aim of these techniques is to **measure material properties during and after the structure formation or structure loss without perturbing the process**. Examples of such processes include gelation, aging (or curing) or temperature-induced changes in dairy products, protein- and polysaccharide-based gels, and food emulsions.

ElastoSens™ X3: Real-time, nondestructive and contactless mechanical monitoring instrument of coagulation, gelation and polymerization kinetics

Most available non-destructive techniques are based on spectroscopic methods, focusing on molecular level characteristics (at nanometer-range distances) and few techniques with the exception of certain rheological methods

© Rheolution Inc. All rights reserved.

Rheolution instruments

(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^{TM} X3 the first 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 couple physical stimuli with mechanical testing.

This application note presents the use of ElastoSens^{TM} X3 in the characterization viscoelastic evolution of common food hydrocolloids.

On ElastoSens^{TM} X3

ElastoSens^{TM} X3 is a benchtop instrument that measures without contact, non-destructively and in real time the viscoelastic properties of gels as function of time and temperature or other stimuli. The patented technological principle behind this instrument is purely mechanical. The sample cup of the instrument is a cylindrical cuvette with 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. Finally, the material response is processed to obtain the viscoelastic properties of the gel (i.e. shear storage and loss moduli). This process is repeated in desired intervals to characterize the mechanical evolution of the studied material.

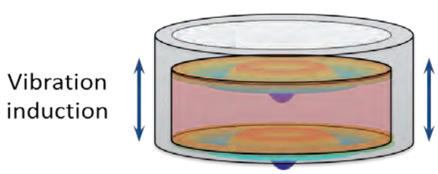

Fig. 1 Principale of ElastoSens^{TM} X3: Non-contact and non-destructive measurement of few micrometers amplitude vibrations.

PROTOCOL AND RESULTS

Agar is a hydrocolloid used as a thermo-reversible gelling agent for foods that can be served cold or hot owing to its high dissolution temperature. It can form gels at very low concentrations and can be used in conjunction with other hydrocolloids to obtain a wide range of textures.

In this work, commercial food-grade agar (Telephone Brand Agar-Agar Powder, Seng Huad Ltd., Thailand) was dissolved in water by heating to boil. A series of concentrations (0.7, 0.5 and 0.2 % w/w) was prepared from a 1 % w/w stock solution. 2 mL of solution cooled to 40 °C was injected in a calibrated sample cup and the evolution of viscoelasticity at 25 °C (set temperature) was followed as a function of time. In another series of experiments, 0.7 % w/w solutions of food-grade agar were prepared and the pH was either kept unchanged (pH 7.4) or adjusted to pH 3.1 by adding a known amount of lemon juice. The solution at pH 3.1 was kept overnight at +5 °C, after which it was reheated to boil and used for measurements as above.

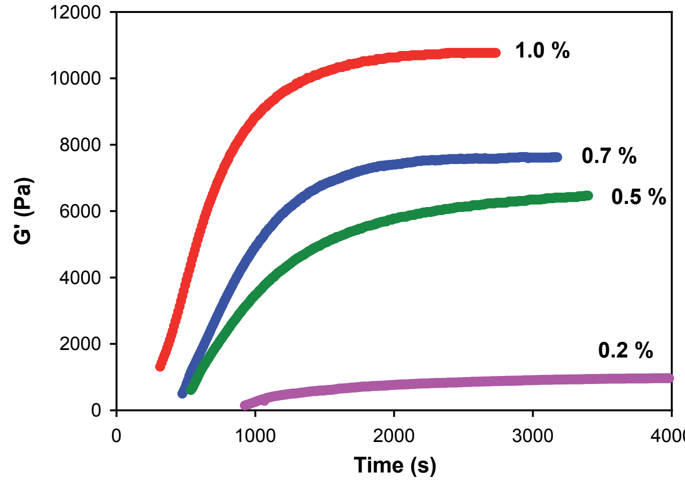

Fig. 2 Gelation kinetics of agar at various concentrations measured at 25 °C by ElastoSens^{TM} X3 (pH 7.4).

Figure 2 shows the concentration dependence of the gelation kinetics of agar at neutral pH and 25 °C. 1 % solution of agar started forming gel after the temperature of the sample dropped from 40 °C to around 30 °C. The gelation of agar occurs via disorder-order transition of polysaccharide chains from random coils to double helices, followed by network formation due to branching of helices, and then by helix bundling. Decreasing the agar concentration leads to slower gelation because of lower probability of association. The gelling temperature and thus the gelation kinetics are also known to be concentration dependent and vary according to the source of agar. When the concentration is decreased to 0.2 %, below the critical overlap concentration (c*) of agar, the association is particularly slow and the resulting gel is weak. The strength of agar gel will decrease over time (storage) because of spontaneous degradation of polysaccharide chains, which also may lead to batch-to-batch variation of gelling properties. ElastoSens^{TM} X3 measures precisely and rapidly the gelation kinetics without altering the structure of hydrogels.

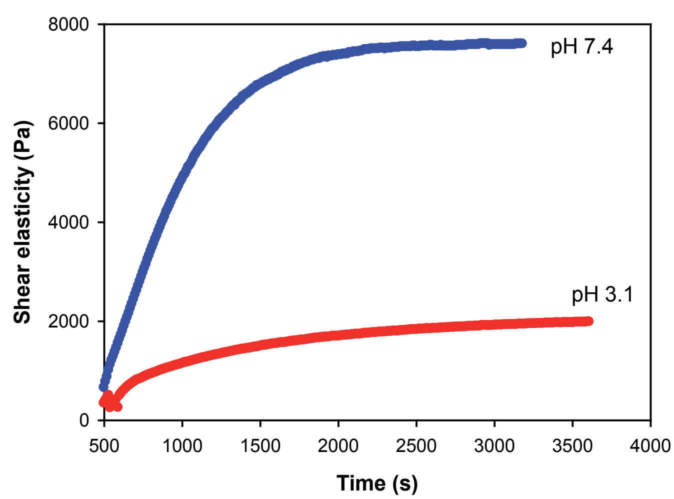

Fig. 3 Gelation kinetics of agar at two different pH values (pH 7.4 and 3.1) at 25 °C by ElastoSens^{TM} X3.

Figure 3 demonstrates the effect of pH on the gelation kinetics and shear elasticity (G') of agar at 25 °C. Storage under acidic concentrations (here pH 3.1) and redissolution by heating leads to hydrolytic degradation of polysaccharide chains. As molar mass affects the gel strength, the degradation will lead to

© Rheolution inc. All rights reserved.

weaker gels and thus lower elasticity. Salts, sugars, polyols (glycerine, ethylene glycol) and other hydrocolloids also modify the strength of agar gels and testing the viscoelastic properties of multicomponent systems is an important part of formulation process.

CONCLUSION AND APPLICATIONS

One of the most interesting applications of ElastoSens^{TM} X3 is the non-destructive real-time measurement of mechanical evolution of various food ingredients and formulations during and after the structure formation (gelation, stabilization, etc.) or structure loss (melting, dissolution, etc.) without perturbing the process. Measurements are conducted without contact and the samples remain sterile. The instrument 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 sample can then be mounted in the instrument at any time and as often as desired to periodically perform new mechanical characterization.

The versatility and unique technical features of ElastoSens^{TM} X3 make it a very useful tool in research and quality control in food and dairy industry. The measuring accessory with three parallel sample holders can significantly improve the statistical significance of measurements. Another version of the technology, CoaguSens^{TM}, allows at-line measurements under processing hall conditions and automated control of processes such as cutting of milk gels at desired curd firmness.

All these capabilities are made possible by the new technology of ElastoSens^{TM} X3. An instrument designed to overcome challenges in research and development in the food and dairy industry.

ElastoSens^{TM} X3

	Specifications of ElastoSens^{TM} X3
Mechanical & Physical	
Shear Elastic Modulus	0 Pa to 100 MPa
Precision	0.1 Pa
Simultaneous measurements	3 samples (optional)
Variable sample volume	YES
Programmable Thermal Rampes	
Temperature range	15° C to 70° C (optional 4° to 70° C)
Precision	0.5° C
Temporal	
Selectable temporal resolution	1 second to 120 minutes
Measurements duration	10 seconds to 1200 hours
Software	
Interface	Simple interface with touchscreen
Software	Test settings, post-processing, real time results display, archiving and data transfer
Automation for QC	
Yes	Contactless measurements

Rheolution Inc.

5333 Casgrain Ave., Suite #712
Montreal, QC, H2T 1X3, Canada

+1 514-586-2006 www.rheolution.com informations@rheolution.com