

## Applied rheology in food technology

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### SYNOPSIS

In addition to oscillation measurements, which are traditionally used to examine the flow behavior of gelling foods, small deformation measurements in CS-mode („controlled stress“) can be carried out to simulate specific application conditions.

In contrast to CR-measurements („controlled rate“), where gel structures are destroyed by shearing, characteristic stress-deformation-profiles can be obtained. Sol-gel-transitions can be investigated by temperature dependant measurements on thermoreversible gelling biopolymers (gelatine).

A temperature dependent compliance  $J(T)$  can be used as a rheological index to describe the gel point temperature  $T_{CP}$ , which can be correlated directly to results obtained from oscillation tests.

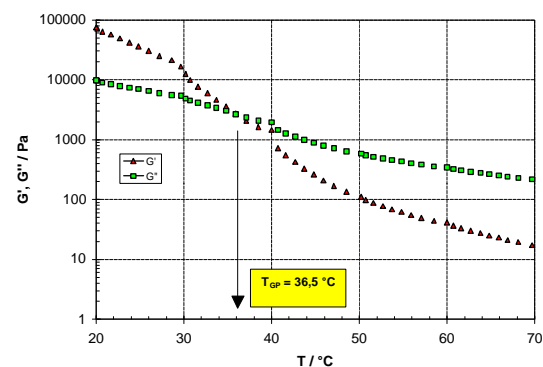
### INTRODUCTION

Paste- and gel-like food products normally exhibit a complex flow behaviour. The reason for this is the strongly heterogenous character of their texture. The rheological properties are influenced by particle structures and occasionally by energetic interactions. This is particularly the case for biopolymer containing systems (e.g. starch, gelatine, xanthane).

By shearing, internal structures of a sample can be destroyed reversibly or even irreversibly. Therefore the characterization in a non-destructive way through oscillation measurements is very helpful. By using these kinds of measurements, kinetics of gelbuilding [1] as well as gel strength [2, 3] may be observed quantitatively. More information per time unit can be collected by using a so called MultiWave-Oscillation, especially for time or temperature dependant structural changes [4]. In order to simulate the application behaviour of food textures it is often desirable to have additional information on the deformation

characteristics caused by shear forces, especially when sol-gel-transitions are supposed to occur.

These are evident for production and application processes while the hot paste product is moved slowly with decreasing temperature (e.g. when filling, feeding, pouring).



*Storage modulus  $G'$  and loss modulus  $G''$  as a function of temperature  $T$  for gelatine (cooling rate:  $5\text{ }^{\circ}\text{C min}^{-1}$ ,  $\omega = 10\text{ rad s}^{-1}$ )*

The stress appearing during these procedures can now be simulated with a controlled stress rheometer.

The aim is to prove how far non-oscillating small deformation measurements supplement the results obtained from oscillating measurements.

### EXPERIMENT

The measurements have been carried out with a controlled stress rheometer HAAKE RS 150 in combination with a Peltier temperature control unit HAAKE TC 80.

The sample (commercial gelatine product) was investigated rheologically using a plate-plate-geometry (diameter 35mm, gap 1 mm).

During the measurements gap control was performed by HAAKE *AutoGap* and *ThermoGap* features.

RESULTS AND DISCUSSION

In order to examine the temperature dependence of the gelation of a sample one may use an oscillating test which describes rheological values (storage modulus  $G'$ , loss modulus  $G''$ , dynamic viscosity  $\eta^*$ ) at a constant angular velocity  $\omega$  (frequency respectively) as a function of temperature.

Figure 1 shows such a measurement for a thermo-reversible gelating gelatine. The temperature was decreased from 70°C to 20°C. The intersection of the storage and loss modulus is often used as a characteristic value (gel point: elastic and viscous portions are equal). To simplify matters one may say that the system is able to flow above the gelation temperature  $T_{GP}$  (dominating viscous properties) and it solidifies below  $T_{GP}$  (dominating elastic properties).

If one takes into account that the viscoelastic properties are highly dependant on the frequency, the determined gelation temperature of  $T_{GP} = 36,5 \text{ }^\circ\text{C}$  may vary with the chosen frequency. In order to get more information on the flowability it is possible to run temperature dependent CS flow curves (controlled stress).

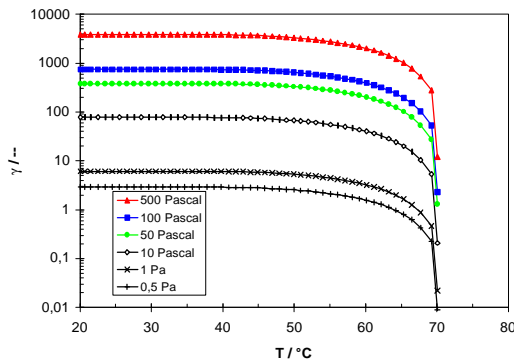


Fig.2: Deformation  $g$  as a function of temperature  $T$  for gelatine at different constant shear stresses (cooling rate:  $5 \text{ }^\circ\text{C min}^{-1}$ )

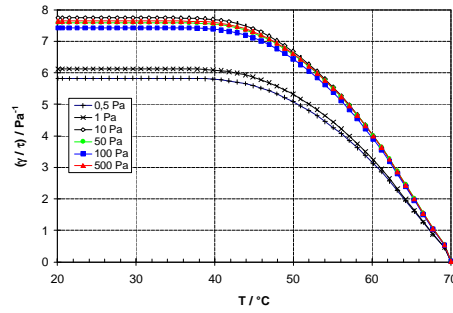


Fig.3: Ratio of deformation  $g$  to shear stress  $t$  as a function of temperature  $T$  [ $=$  compliance  $J(T)$ ] for gelatine (cooling rate:  $5 \text{ }^\circ\text{C min}^{-1}$ )

Figure 2 shows measurements where several constant values of shear stress have been applied. The resulting curves exhibit a specific shape.

The resulting curves exhibit a specific shape. At high temperatures (above  $T_{GP}$ ) the deformation increases rapidly. With decreasing temperature the raise in deformation is increasingly slower, until, when the gel state is reached (below  $T_{GP}$ ), no movement can be detected any more.

A standardization of these curves can be performed by defining a temperature dependent compliance analogous to creep measurements. Therefore we calculate the quotient of deformation and shear stress:

$$J(t,T) = \frac{g(t,T)}{t}$$

This has been done for the examined gelatine and is shown in figure 3. It can be seen that the compliance is independent of applied stress for medium shear stresses. For extreme low stresses the compliance is lower.

The gel point temperature  $T_{GP}$  can be calculated from the point when the compliance does not change any more with decreasing temperature. The results are presented in table 1.

For stresses between 10 Pa and 500 Pa there is an excellent agreement for the calculated gel point temperature  $T_{GP}$ . For very low stresses both the measured compliance is lower and the gel point temperature  $T_{GP}$  is higher.

Applied stress $\tau$	$T_{GP}$
0,5 Pa	38,75 °C
1 Pa	38,76 °C
10 Pa	37,76 °C
50 Pa	37,75 °C
100 Pa	37,76 °C
500 Pa	37,74 °C

Table 1:  
Gel point temperatures  $T_{GP}$  calculated from CS-temperature-dependent measurements for different stresses

This means that at this temperature a gel structure is present which can be destroyed using methods with higher stresses (including oscillation temperature ramps, where usually a broad range of stresses is required).

## CONCLUSION

When processing gelling food products, different types of stresses usually are applied. Considering the variation of temperature during the process typical sol-gel-transitions may occur.

	Prep.+ Transport	Filling	Forming
Application	dispensing, heating, pumping	feeding, pouring, die moulding	cooling, structuring
Temperature	above gel point temperature $T_{GP}$	around gel point temperature $T_{GP}$	below gel point temperature $T_{GP}$
Texture	sol	sol-gel- transitions	gel
Rheology	shear flow measurements	contr. stress measurements	oscillation measurements
Index	$\eta(\dot{\gamma})$	$J(t,T)$ $\gamma(t,T)$	$G', G'', \tan \delta$ $\eta^*, (\omega, \tau, \dot{\gamma})$

Table 2:  
Relationship between application and rheology for food gels in processes.

CS measurements are suitable to detect these effects „close to application conditions“.

In table 2 a general relationship between application parameters and rheological indices is presented

Moreover, these results clearly show that the gel point is no „point“ although rheological treatments imply this. Actually, the transition

from sol to gel state covers a certain temperature range where an ordered three-dimensional structure is formed.

## LITERATURE

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