

Modeling of the SAG Test for Pectin Gels

Heidi Nielsen^{1,3}, Mette Kolte², Beinta Unni Nielsen³, and Søren Hvidt¹

¹Department of Chemistry, Roskilde University, DK-4000 Roskilde, Denmark

²Department of Chemical Engineering, The Technical University of Denmark (DTU), DK-2800 Lyngby, Denmark

³Copenhagen Pectin A/S, DK-4623 Lille Skensved, Denmark

ABSTRACT

The relationship between the height of a gel determined by a sag test and the elastic modulus (G') has been simulated using a finite element analysis. The sag of pectin gels with varying pectin concentrations and the corresponding G' values were measured. The obtained sag results are compared with the predictions from the simulations. The agreement between the predictions and the experimental data is good, and it shows that the deformation can be modeled accurately using a model based on an incompressible elastic material.

INTRODUCTION

Pectin is a polysaccharide, which is widely used in the food industry as a gelling agent.¹ In 1959 the American Pectin Committee finished several years of research resulting in the development of the SAG test for determining the grade strength of pectins irrespective of origin.² This method is based on measuring the sag, which is the height decrease of an inverted pectin gel made in glasses of very precise dimensions. A ridgelimeter is used to determine the percent sag.²

The SAG method fails to give information about gel strength for many pectin applications, since it only describes the pectin grade at one well defined condition (65% soluble solids at pH 3).² The measured sag is slightly dependent on time, and it is determined two minutes after inversion of the gel in the recommended SAG test.² The SAG

values only serve as standardization parameters for pectins, and it seems desirable to link the SAG value to more fundamental rheological properties of the gels.

Experiments presented below show that pectin gels are nearly ideal soft elastic systems in the time range of interest. This observation suggests that the gel can be modeled as an incompressible elastic material.

The deformation, which is due to gravity, is inhomogenous. The top part of the gel is expected to be deformed less than the interior and lower parts of the gel. In order to determine the deformation in various parts of the gel a finite element analysis has been used. The calculations result in a knowledge about the shape and the sag of a gel with the gel geometries used in the standard SAG test. We report here results of such simulations, which correlate the sag with fundamental properties of the gel. The results of the simulations are furthermore compared with experimental sag and rheological results.

MATERIALS AND METHODS

Pectin solutions were prepared following a standard procedure, and poured into standard SAG glasses.² A GENU pectin type B rapid set from Copenhagen Pectin was used to make gels with pectin concentrations between 6 and 10 g/L.

The standardized SAG glasses have bottom and top diameters of 39.7 ± 0.5 mm and 63.5 ± 0.3 mm, respectively, and a height, H_0 , of 79.4 ± 0.2 mm. The gels were allowed to form at 25 °C for approximately 24 hours.

the slip and no-slip conditions are shown in Fig. 1 for the same value of $\rho g H_0 / G$. As expected, it is seen that the relative height decrease is greater for the slip condition.

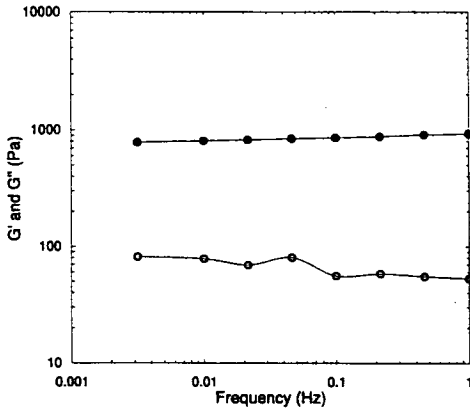


Figure 3. Plot of the frequency dependence of G' (filled circles) and G'' (open circles) for a gel containing 9 g/L pectin at a stress amplitude of 2 Pa.

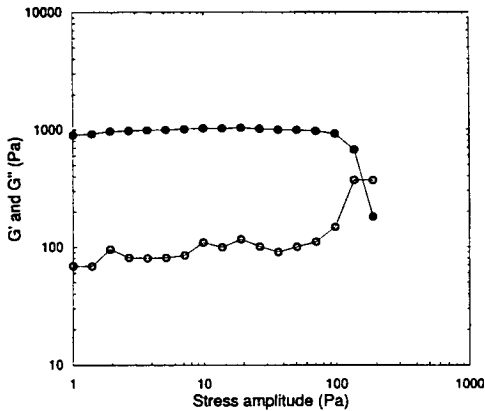


Figure 4. Plot of G' (filled circles) and G'' (open circles) against stress amplitude for a gel containing 9 g/L pectin at 1 Hz.

Simulations were carried out for a range of relevant $\rho g H_0 / G$ values. The sag results are summarized in Fig. 2 for both slip and no-slip conditions. A slightly curved relationship between sag and the dimensionless factor is

observed. The figure illustrates that a small sag should be expected, when the density is low or the elastic modulus is large.

The elastic shear modulus, G' , of pectin gels is almost independent of frequency between 0.003 and 1 Hz, as shown in Fig. 3. Furthermore the gels are in the linear elastic region up to stress amplitudes of 40 to 100 Pa for gels containing from 6 to 10 g/L of pectin, respectively, as illustrated in Fig. 4. G' values for a number of such gels varied between 200 and 2200 Pa, and the density measurements showed an average density of 1.30 ± 0.01 g/cm³ for all the gels.

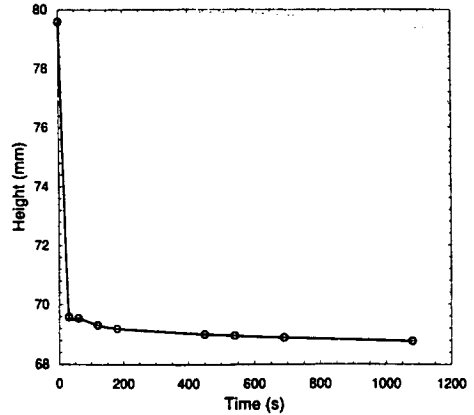


Figure 5. The height of an inverted gel (9 g/L pectin) as a function of time.

The heights of the pectin gels were measured as a function of time after the gel was placed on the glass plate. A typical result is shown in Fig. 5. The gels undergo a rapid initial decrease in height followed by a slow decrease at longer times, as expected for a gel with little frequency dependence as shown in Fig. 3. The two minutes setting time used in the standard SAG test is in a time range where the height varies slowly with time.

When a slip model was assumed the softest gels were predicted to slide to a diameter of more than 80 mm. However, the diameters of the inverted gels were found experimentally to be approximately 70 mm independent of pectin concentration (data not shown). This indicates that the gels follow the no-slip model.

The time dependence of the sag as well as the rheological measurements indicate, that the gels are primarily elastic. This assumption,

The densities of the gels were measured gravimetrically.

A ridgelmeter was used to measure the height of the deformed pectin gels. After the height was measured, a slice of the gel was cut with a thickness of 3 mm, and it was transferred to a Haake rheometer (RS100) using parallel plates with a diameter of 35 mm. The elastic and loss shear moduli, G' and G'' , were measured as a function of frequency (0.003 to 1 Hz, at 1-2 Pa) and stress amplitude (1 to 300 Pa, 1 Hz) at 25 °C.

THEORY AND SIMULATIONS

The simulations were performed by finite element analysis as described elsewhere.³ An initial undeformed mesh with dimensions of half of the axisymmetric SAG glass was used (height, H_0 , 79.6 mm, radius, R_1 and R_2 , 20.8 and 32.0 mm, respectively).

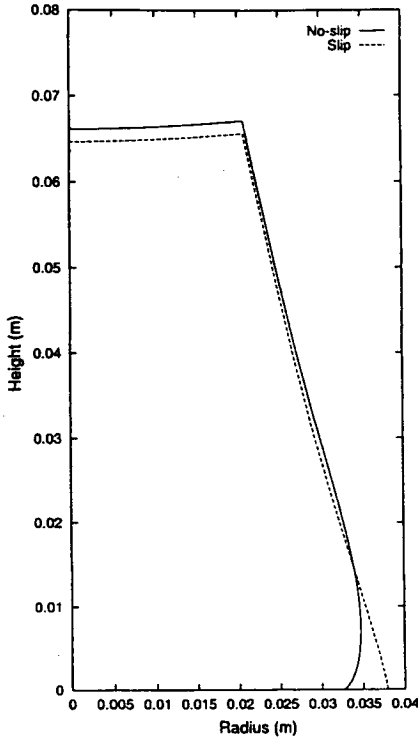


Figure 1. Contours of a sagged gel at $\rho g H_0 / G = 1.759$ for both slip (broken line) and no-slip conditions (solid line). The heights of the gel surfaces are plotted against radius.

It was assumed that the material is an ideal elastic material, obeying the law for a Hookean solid with proportionality between stress and strain, $\sigma = G\gamma$. The only external force acting on the gel is due to gravity, g . We have defined the sag as a dimensionless variable, $\Delta H / H_0$, where ΔH is the change in height after the gel has sagged. The sag depends on the density of the gel (ρ), gravity (g), and the elasticity of the material. A dimensional analysis suggests that $\Delta H / H_0$ is a function of the dimensionless variable, $\rho g H_0 / G$, where G is the elastic modulus at zero frequency. In practice the elastic modulus used in comparison with simulation results was measured on the rheometer at 0.01 Hz.

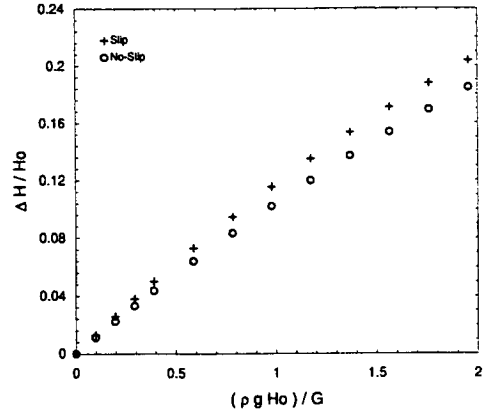


Figure 2. Simulated sag, $\Delta H / H_0$, plotted against $\rho g H_0 / G$ for both slip (+) and no-slip (o) conditions.

The simulations were performed using two different models: a slip simulation, where the gel is allowed to spread on the plate when the gel is inverted onto the glass plate; and a no-slip simulation, where the bottom mesh elements are fixed in place, i.e. the gel is not allowed to change its diameter on the plate. The simulations involve solving the equations of motion for each element defined by the mesh.

RESULTS AND DISCUSSION

The contours of the sagged gels varied depending on the model used in the simulations. The contours of gels following

which is used in the simulations, seems therefore justified.

The experimental data and the simulated predicted relationship are compared in Fig. 6. It is interesting to see that the sag determinations at each of the four concentrations are in excellent agreement. This demonstrates that the SAG test is highly reproducible for the four concentrations investigated. The measured G' values, however, vary significantly at each concentration. The large variation in the rheological data is probably due to problems cutting the gel slices, which were loaded into the rheometer. It is difficult to cut the gel into disks with a uniform thickness, and the surfaces of the slices appear rough, especially at higher pectin concentrations. The fixed gap of 3.00 mm may therefore very likely squeeze the gels differently.

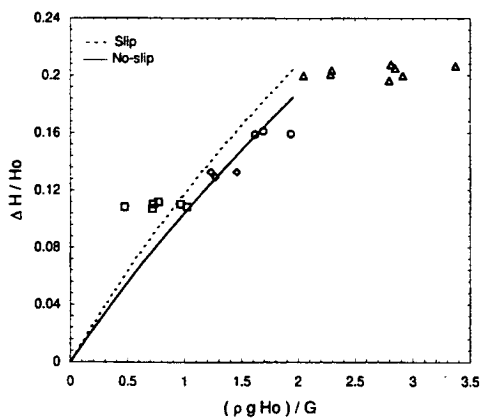


Figure 6. Experimental sag and G' data combined with the simulated predictions (curves). Several data points are shown for each pectin concentration in g/L: 6 (triangles); 7 (circles); 8 (diamonds); 10 (squares).

Despite the scatter in the data it is reassuring to see the good agreement between the simulated and measured sag. The simulations are able to predict the right order

of sag, and it demonstrates that the sag can be modeled quite accurately using the simple incompressible elastic model. Furthermore the results in Fig. 6 show that the experimental data are closest to the no-slip simulation predictions except for the 10 g/L data. More direct support for the no-slip model was, as mentioned above, the constant diameter of the gel on the plate.

CONCLUSION

The SAG test is very attractive and practical, since it gives highly reproducible results and is fairly simple to use without the need for expensive instruments. Our simulations demonstrate that the sag numbers can be predicted theoretically from measured values of the elastic shear modulus and the density of the gels. The measured diameters of the inverted gels indicate that the gels stick to the glass plate and a no-slip simulation model gives the most accurate predictions of the sag numbers. The simulations presented here are not restricted to pectin gels, but can be used to predict sag values for any incompressible elastic gel knowing its shear modulus and density.

ACKNOWLEDGMENT

Ole Hassager and Peter Szabo are thanked for many valuable discussions and help.

REFERENCES

1. Thakur, B.R., Singh, R.K., and Handa, A.K. (1997), "Chemistry and uses of Pectin - A Review", *Critical Reviews in Food Science and Nutrition*, **37**, 47-73.
2. Baker, G.L. (1959) "Pectin Standardization - Final Report of the IFT Committee", *Food Technology*, **13**, 496-500.
3. Rasmussen, H.K. and Hassager, O. (1995), "Simulation of Transient Viscoelastic Flow with Second Order Time Integration", *Journal of Non-Newtonian Fluid Mechanics*, **56**, 65-84.