

Comparison of Rheological Properties of β -lactoglobulin / Potato Amylopectin Gels in Dynamic Shear and Dynamic Compression.

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ABSTRACT

Oscillating measurements in shear were compared with oscillating measurements in compression for a particulate β -lactoglobulin gel filled with potato amylopectin of different concentrations and viscosity.

INTRODUCTION

There is a great interest in understanding how the structure of complex food products is correlated with the texture. The structure at different levels, e.g. molecular and colloidal levels, may correlate with different rheological behaviours or methods.

There is sometimes no relation between small and large deformations or between different small deformation techniques¹. Differences in the rheological properties do not always correspond to differences in the microstructure and vice versa².

β -lactoglobulin forms gels on heating, and forms a particulate network in the vicinity of the isoelectric point, which is around 5.2, for β -lactoglobulin. The particulate gel network is composed of almost spherical aggregates linked together to form the threads of the network³.

Potato amylopectin does not form gels. Weak gels may perhaps be formed after long times at high concentrations. It is possible to achieve high viscous and clear solutions of potato amylopectin without the typical starch structure consisting of swollen granules. Instead, it forms a macromolecular solution⁴.

The purpose of this work was to compare different rheological methods in an investigation of the rheological alterations occurring when a particulate protein gel (β -lactoglobulin) is filled with a viscous solution (potato amylopectin).

MATERIAL AND METHODS

Materials

The β -lactoglobulin samples used, WPI PSDI-2400, were obtained from MD Foods ingredients, Denmark. The content of the product was: 92% protein, of which 95% was β -lactoglobulin, less than 1% fat, less than 0.2% lactose and 5.5% moisture. The granular amylopectin potato starch (pap) was kindly supplied by Lyckeby Stärkelsen, Sweden. The potato used for this was developed using genetic engineering to suppress amylose synthesis. The amylose content was 2% maximum, the ash content 0.5% and the moisture content 20%. (Dry weight of pap and β -lactoglobulin was used in the experiments.)

Sample preparation

Pap and distilled water was mixed and warmed. To obtain a molecular solution of pap, it was necessary to warm the dispersion of granules to 120°C. The solution was then cooled in room temperature to 20°C. The β -lactoglobulin was mixed into a solution of distilled water. The pH was adjusted to 5.4 with 0.5M HCl. The pap solution was then

mixed with the β -lactoglobulin solution. The samples had a constant β -lactoglobulin concentration of 6%(w/w) and pap concentrations of 0, 0.25, 0.5, 0.75, 1.0 or 2.0%(w/w). To obtain a lower molecular weight of the pap, the solution was sheared in a homogenizer, Ultra Turrax, T25, (JANKE & KUNKEL IKA®-labortechnik) at a rate of 20500 min⁻¹ for 1 min, before mixing it with the β -lactoglobulin solution. The pH was checked, and the samples were then degassed.

The samples were poured into moulds made of aluminium. The moulds were cylindrical in shape with open ends. They had an inner diameter of 15mm and a length of 60mm. The ends were closed with rubber stoppers at the bottom and with heat-proof tape (scotch 425) at the top. The cylinders had been greased with bearing grease to prevent the samples from sticking to the mould. The cylinders were then placed into a FP40-MS Julabo programmable silicon oil bath (Julabo Labortechnik GmbH/Seelbach, Germany). The temperature was increased to 90°C at a heating rate of 2.5°C/min and held at 90°C for 1h. Thereafter, the temperature was decreased to 20°C at a cooling rate of 2.5°C and held at 20°C for 1h.

Measurements

The gels were cut into 5mm high cylindrical test pieces for oscillatory measurements in shear with the parallel plate system. The measurements were performed with the Bohlin VOR rheometer (Bohlin Rheology, Chichester, UK) with a 15mm plate, a frequency of 1 Hz, a strain of 8×10^{-4} and a torsion bar of 3.5gcm. A thin filter paper with a diameter of 15mm was placed on both plates to prevent slip during measurements. The measurements of the complex modulus, $|G^*|$, were performed after a time sweep of 180s. The moduli were constant.

Oscillatory measurements in compression were performed by DMA, Dynamic Mechanical Analysis, in a Rheometrics RSA-

II (Rheometrics Scientific, Piscataway, NJ, USA). The test pieces were cut to 20mm long cylinders and glued to plates covered with sandpaper, using a cyanoacrylate glue, Loctite 401. The diameter of the plates was 15mm, with a frequency of 1Hz and a strain of 4×10^{-3} . The measurement of the modulus was made after a time sweep of 180s. The height to diameter ratio was 4/3 to avoid the influence of squeeze flow. The modulus slowly increased during the measurement, probably owing to the self-compression of the test piece.

RESULTS AND DISCUSSION

The properties of the mixed gels have been studied by two different techniques of small deformations, one technique measuring the complex modulus in shear, and one technique measuring the complex modulus in compression. Both complex moduli, $|G^*|$ and $|E^*|$, were obtained from oscillatory measurements in a parallel plate measuring system.

Fig. 1 shows modulus $|G^*|$ and modulus $|E^*|$, measured with the parallel plate technique. The values of $|G^*|$ and $|E^*|$ for a pure β -lactoglobulin gel are shown as a straight line. The curves measured up to a pap concentration of 2.0% show $|G^*|$ and $|E^*|$ for gels containing sheared pap, i.e. with a low molecular weight, and the curves measured up to a pap concentration of 0.75% show $|G^*|$ and $|E^*|$ for gels containing unsheared pap, i.e. with higher molecular weight. Gels containing unsheared pap at concentrations higher than 0.75% were so weak that no measurements could be performed.

The $|G^*|$ and $|E^*|$ curves corresponded very well to each other (Fig. 1). The values obtained in compression can be compared to the values obtained in shear by:

$$E = 2(1 + \nu)G \quad (1)$$

where ν is Poisson's ratio. If the material is elastic and incompressible, i.e. the volume of the material is constant, Poisson's ratio can be set to $\nu=0.5$ and equation (1) is reduced to $E=3G$. The measured $|E^*|$ values were about 4-6 times the measured $|G^*|$ value, and the ratio of $|E^*|/|G^*|$ was higher the higher the values of the modulus. Comparisons of the values obtained from the modulus of deformability with the shear modulus have been performed on cheese samples^{3,6}. The calculated E/G^* ratios there were also higher than 3, i.e. in the range of about 3.5-6.

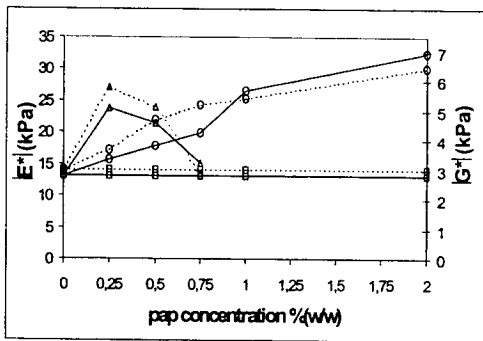


Figure 1. $|G^*|$ and $|E^*|$ versus potato amylopectin concentration for mixed gels containing 6%(w/w) β -lactoglobulin and 0-2%(w/w) potato amylopectin, (Δ), pure β -lactoglobulin, (Δ), unsheared pap, (O), sheared pap, (---), compression measurements, (—), shear measurements).

At low pap concentrations, where the pap had a small influence on the β -lactoglobulin, the $|G^*|$ and $|E^*|$ increased.

At higher pap concentrations, there was a large difference between gels containing sheared and unsheared pap. Here, the protein network formation was strongly influenced by the high molecular weight of pap.

The gels were studied by light microscopy (LM) and it could be seen that the β -lactoglobulin network became more open the higher the concentration and the higher the molecular weight of the pap.

The pap had a great influence on the modulus. This increase in modulus for gels containing sheared pap is convincing, since pure pap has a modulus of less than 1 Pa. The pores in the β -lactoglobulin network also became larger the higher concentration of pap, and still the modulus increased!

$|G^*|$ and $|E^*|$ were also measured with the parallel plate system with a diameter of 30mm, not shown here. This gave the same results as for the 15mm plate.

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