# Gelatinisation of Potato Starch, as Followed by Simultaneous Light Microscopy and Small Deformation Oscillation Rheology

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ABSTRACT

A newly developed instrument, the ReoVision, that combines a controlled strain rheometer with a conventional light microscope was used to follow the heat induced gel formation of potato starch. A description of the instrument is given, as well as some early results.

## INTRODUCTION

The gelatinisation of starch can be described as *"... the collapse (disruption) of molecular orders within the starch granule manifested in irreversible changes in properties such as granular swelling, native crystallite melting, loss of birefringence, and starch solubilization. The point of initial gelatinization and the range over which it occurs is governed by starch concentration, method of observation, granule type, and heterogeneities within the granule population under observation."*[1].

Numerous methods can be used to follow the gelatinisation, each giving a description that contributes to the overall understanding of the process. Among frequently used techniques, light microscopy[12], differential scanning calorimetry[4,11,15], x-ray crystallography[16,17] and nmr[6,13], as well as empirical[14] and fundamental[2,5,7] rheological measurements can be mentioned.

In the food industry, the Brabender Amylograph is a *de facto* standard for starch quality determination. In the last decade, fundamental rheological techniques have also become more and more in use, however more in pure research than in quality control. It is well known that starches of different origins do not follow the same gelatinisation pattern [3]. Not only the temperature interval for the gelatinisation, but also the swelling and disruption patterns of the starch granules differ, both between and within species, and as a consequence, the structure of the resulting gel or paste is also species dependent.

Fundamental rheological studies of starch gelatinisation require a controlled strain instrument, as the increase in the viscosity is to fast for the simulated controlled strain facilities of most controlled stress instruments. Further, the instrument must be equipped with a sealed cell, to avoid evaporation of water and surface film formation. Sedimentation of the starch granules can be avoided by the use of a carrier system, a gel or a solution with a sufficiently high viscosity.

Light microscopy is a simple method to follow starch gelatinisation. The equipment needed is a microscope, equipped with a hot stage, and preferably with polarisation filters, as the non-gelatinised starch granules are birefringent. Staining with markers such as iodine, Congo red, etc., is

also frequently used[8,9,10].

The properties of a starch paste depend very much on its thermo-mechanical history, a fact that makes it difficult to directly compare data from various experiments, where different methods are used. The forces applied may differ between the methods, and equally, the heat transfer characteristics may differ from one instrument to another. We have therefore collaborated with Bohlin Reologi[18] on the construction of an instrument where small deformation oscillatory rheological measurements can be performed on a sample that is simultaneously monitored by a light microscope.

## REOVISION

The newly designed ReoVision is a controlled strain instrument. The measurement cell has a fixed 75 mm plate-plate geometry, with a variable gap. Glass windows in the upper and lower plates allow the combination with optical measurement techniques.

Our instrument is equipped with a sealed cell, that allows high temperature experiments on water based systems like starch hydrogels. The temperature is controlled by circulating water, that heats or cools both the sealed cell and the bottom plate. A water reservoir inside the sealed cell prevents the sample from drying. A microscope tube with a CCD video camera is fitted above the glass window, and below the glass window of the bottom plate, a light source is connected through an optical fibre.

The rheometer is controlled from a specially developed Windows-based software, that also handles data from the rheometer and images from the microscope.

## STARCH GELATINISATION

We have used the ReoVision for some preliminary experiments, where the gelation of potato starch in some different carrier gels has been monitored. Only one frequency, 1Hz, was used, and the gap between the plates was set to 0,15 mm.

The temperature program was an adapted version of the standard program used in the Brabender Amylograph, *i. e.* a temperature ramp from 45 to 90 °C, at 2°C per minute, followed by a holding period of 15 minutes, and finally a temperature drop back to 45°C, also by 2 °C per minute. The interval between the measurement points was 30 seconds during the heating, and 100 seconds during the hold and cooling periods. Micrographs were taken at relevant intervals.

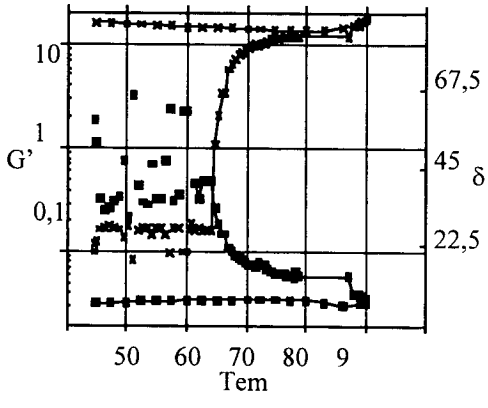


Figure 1. Gelation of 3,5% (w/v) potato starch dispersed in a 1,5% (w/v) paste of the same starch. Symbols ♦ represent the phase angle (δ), symbols x the elastic modulus (G') in Pa.

Figure 1 shows an example of a results graph, from an experiment with a dispersion of 3,5% dry potato starch in a carrier gel made of a 1,5% paste of the same starch. Some more examples, with corresponding micrographs are shown at the Conference.

## DISCUSSION

The combination of rheology and microscopy has the potential to add to our

understanding of the gel formation, not only of starch, but also of other gelling agents, *e.g.* polysaccharides and proteins. However, the interpretation of the micrographs needs improvements, possibly by the use of image analysis software. Further, the rheological characterisation would be more complete if frequency sweeps could be performed more easily. This would, however, require an extended experiment time.

The particular process attempted here, the gelatinisation of starch, requires the carrier gel system to be optimised, so that the interaction with the gelatinising starch is minimised. Also, the staining of the starch could be improved, so detailed images of the starch pastes at high temperatures can be available.

Below the onset temperature of the gelling, the system is also below the sensitivity range of the instrument, but above 65°C, the different stages of the gelation of starch can easily be distinguished, as seen in Fig. 1. The association of molecules is finished during the holding period at 90°C, and is followed by a decrease in the elastic modulus, mainly a heat effect. The setting of the paste during cooling is seen as an increase in the elastic modulus, whereas the phase angle remains constant.

The onset temperature is significantly higher than the onset temperature of the gelatinisation, as measured by dsc or polarisation microscopy. There are differences in the heat transfer, but the main reason for this difference is that the dsc measures the melting of crystallites within the starch granule, prior to the swelling of the granules, whereas the gelation, *i. e.* the association of exudated material from the swollen granule, takes place much later.

For specific applications, the light microscope might have to be replaced by other techniques, such as fluorescence microscopy, or laser light scattering. With the modular construction of the ReoVision, this should be easily accomplished.

REFERENCES

1. Atwell, W. A., L. F. Hood, E. Varriano Marston and H. F. Zobel. (1988). "The Terminology and Methodology Associated with Basic Starch Phenomena." Cereal Foods World., 33, 306-311.

2. Bohlin, L., A.-C. Eliasson and T. Mita. (1986). "Shear Stress Relaxation of Native and Modified Potato Starch Gels." Starch/Stärke., 38, 120-124.

3. Bowler, P., M. R. Williams and R. E. Angold. (1980). "A Hypothesis for the Morphological Changes which Occur on Heating Lenticular Wheat Starch in Water." Starch/Stärke., 32, 186-189.

4. Eliasson, A.-C. (1980). "Effect of Water Content on the Gelatinization of Wheat Starch." Starch/Stärke., 32, 270-272.

5. Evans, I. D. and D. R. Haisman. (1979). "Rheology of Gelatinised Starch Suspensions." Journal of Texture Studies., 10, 347-370.

6. German, M. L., A. L. Blumenfeld, Y. V. Guenin, V. P. Yuryev and V. B. Tolstoguzov. (1992). "Structure Formation in Systems Containing Amylose, Amylopectin and their Mixtures." Carbohydrate Polymers., 18, 27-34.

7. Janas, P. (1991). "Rheological Studies on Potato Starch Pastes at Low Concentrations Part III: Simplified Measurements of Absolute Characteristics of Pasting." Starch/Stärke., 43, 168-171.

8. Padmanabhan, S. and M. Ramakrishna. (1993). "Methods for Estimation of Starch - A Critical Appraisal." Journal of Food Science and Technology - Mysore., 30, 313-320.

9. Revilla, M. A., D. Tolivia and J. F.

Tarrágo. (1986). "A New and Permanent Staining Method for Starch Granules using Fluorescence Microscopy." Stain Technology., 61, 151-154.

10. Schoch, T. J. and E. C. Maywald. (1956). "Microscopic Examination of Modified Starches." Analytical Chemistry., 28, 382-387.

11. Stevens, D. J. and G. A. H. Elton. (1971). "Thermal properties of the starch/water system. I. Measurement of heat of gelatinisation by differential scanning calorimetry." Starch/Stärke., 23, 8-11.

12. Svegmark, K. and A. M. Hermansson. (1991). "Distribution of amylose and amylopectin in potato starch pastes: effects of heating and shearing." Food Structure., 10, 117-129.

13. Welsh, E. J., J. Bailey, R. Chandarana and W. E. Norris. (1982). "Physical characterisation of interchain association in starch systems." Progress in Food and Nutrition Science., 6, 45-53.

14. Windhab, E., G. Tegge and U. Tiemeier. (1988). "Absolutmessung des rheologischen Verhaltens von verkleisterten Stärken." Starch/Stärke., 40, 20-24.

15. Yuan, R. C., D. B. Thompson and C. D. Boyer. (1993). "Fine structure of amylopectin in relation to gelatinization and retrogradation behavior of maize starches from three wx-containing genotypes in two inbred lines." Cereal Chemistry., 70, 81-89.

16. Zobel, H. F., S. N. Young and L. A. Rocca. (1988). "Starch gelatinization: an X-ray diffraction study." Cereal Chemistry., 65, 443-446.

17. Svensson, E. "Crystalline Properties of Starch", Thesis. Lund University, 1996.

18. Bohlin Reologi AB, Öved, Sweden