

Gelation Profiling of Cold Setting Pectin in Confectionery Systems Activated by Controlled Acidification

Niall W.G. Young^{1,2}, Ole Tarp Madsen¹, Henrik Aabye Jensen¹, and Flemming Møller¹

¹ Danisco A/S, Edwin Rahrs Vej 38, DK 8220 Brabrand, Denmark

² University of Chester, Environmental Quality and Food Safety, Parkgate Road, Chester CH1 4BJ, U.K.

ABSTRACT

Gelation of high ester (HE) pectin has been shown to occur under cold conditions through controlled acid release via Glucono Delta Lactone (GDL). Simultaneous rheological and pH profiling allows prediction of the gelation properties to be made for given confectionery systems. Lissajous figures confirm the transition from sol to gel, and verification of the gelation process is obtained via turbiscan measurements.

Increasing temperature showed a reduction of the time required to achieve onset of gelation, and pointed to a new gelation mechanism for HE pectin. Under cold conditions hydrophobic interactions are formed exclusively before acid addition takes place. As temperature is increased in the presence of acid both hydrophobic and hydrogen bonding occur simultaneously as they do under normal hot gelation conditions.

This novel gelation of pectin has seen commercial application in the UK confectionery market.

INTRODUCTION

Traditionally wine gums with high soluble solids and low pH made from high

ester (HE) pectin gel at high temperatures. Any gelation of pectin by controlled proton release has focused on low ester pectin^{1, 2}. Certain applications cannot utilise high temperature gelation due to process restrictions, e.g. direct moulding into chocolate cups. Controlled acidification was investigated with high ester pectin and gelation occurred over time at ambient temperature.

MATERIALS AND METHODS

GRINDSTED® Pectin CF 130B was used together with the acidifier, Glucono Delta Lactone (GDL) throughout the experiments. Samples were analysed by controlled stress rheology, turbiscan and simultaneous pH monitoring.

RESULTS AND DISCUSSION

Figure 1 indicates how long the system is workable after the addition of GDL at time zero and before the onset of gelation, indicated by the downturn of the phase curve; and onto the setting point depicted here as G' / G'' crossover. This represents important parameter diagnostics for predicting the functionality of the cold set system.

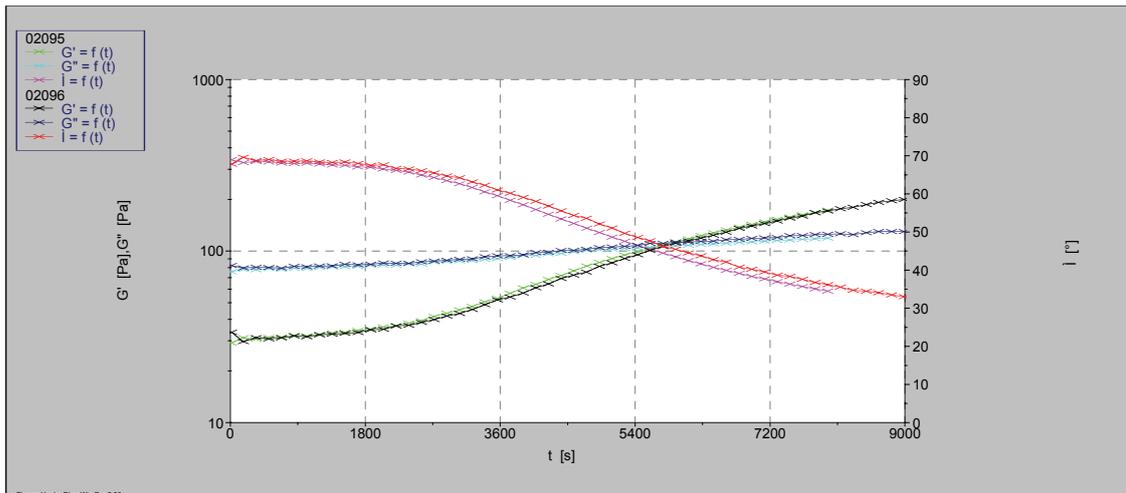


Figure 1 – Gelation profile of GRINDSTED® Pectin CF 130B at 2% GDL addition and 23°C.

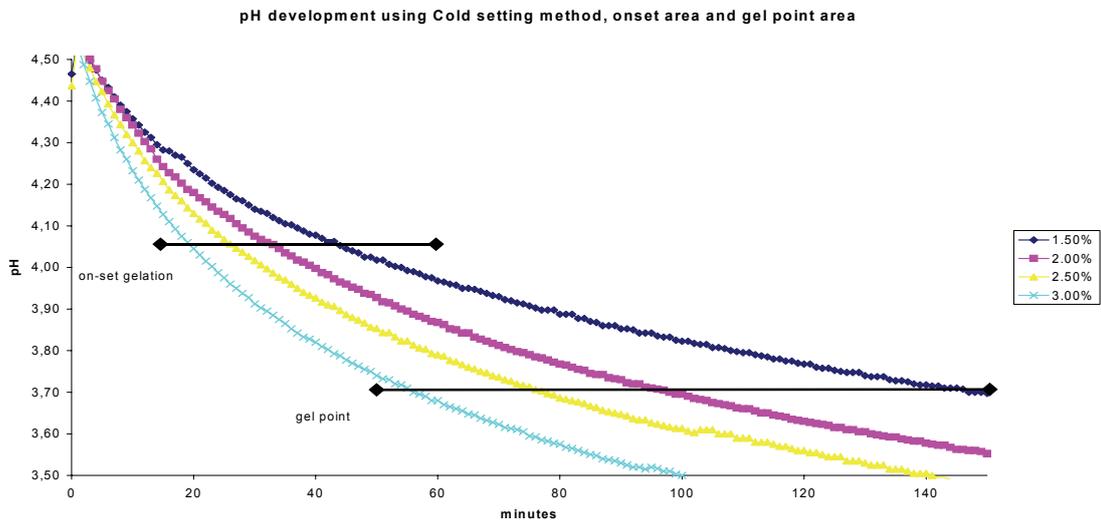


Figure 2 Averaged pH curves for varying acidifier dosages indicating the onset of gelation and gel point.

Figure 2 shows the data of simultaneous pH measurements over four different GDL concentrations, where the results for 2% curve agree well with Figure 1 above – also with 2% GDL. This simple pH measurement can be used to follow and monitor the gelation profile instead of rheometry, once the initial gel profiling has been characterised. This allows easy translation of the technique into industry, where rheometry is not always available

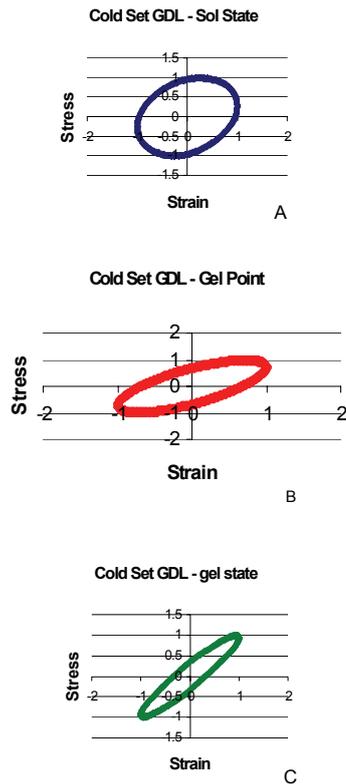


Figure 3, (A-C) shows Lissajous plots of the various states of the cold set pectin system from the sol state (a), Gel point (b), and gel state (c).

The three Lissajous plots show the characteristic change with increasing structure, i.e. a move away from circular to elliptical curves. Figure 3a is from the sol state, corresponding to high pH, Figure 3b is at the gel point, pH 3.7; and Figure 3c is in the gelled state at pH <3.7. The Lissajous figures confirm the gel transition, where the sample is seen to progress from broadly circular to elliptical forms of Lissajous. However, even in what is called the sol state, structure is apparent since the profile is not truly circular.

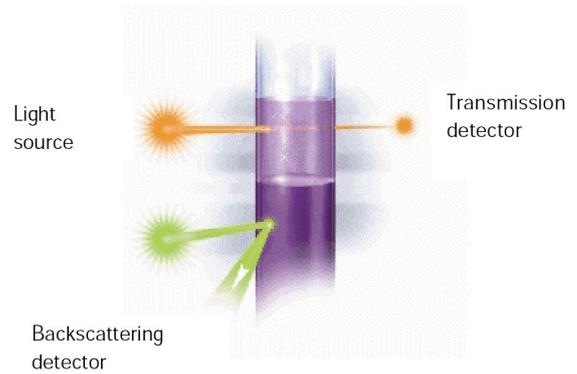


Figure 4 Schematic of the principles of turbiscan.

Non-invasive static light scattering / transmission spectroscopy (turbiscan)³ was used to try and verify the times for gelation from Figure 1. The principles are schematically shown in Figure 4.

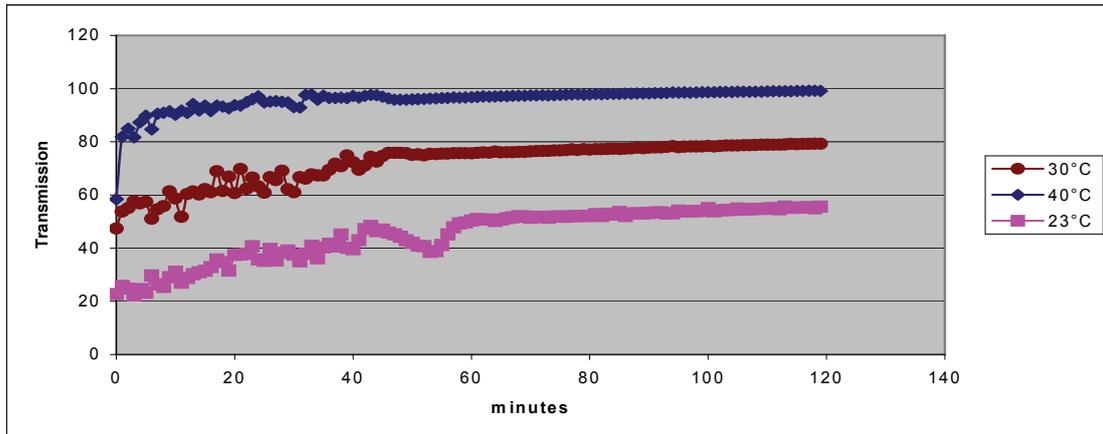


Figure 5 Transmission vs. time for one pectin sample over three different temperatures.

The initial noise in Figure 5 is attributed to air bubbles moving in the non-gelled sol state, which stop once the sample gels. This is seen as the onset of the smooth region, similar to the principles described by Mielke and Dunstan⁴.

The smooth region for 23°C appears to start at ~60 minutes, whereas the G' / G'' crossover in Figure 1 occurs after some 90 minutes. This suggests that sufficient structure was present after 60 minutes to stop the air bubble, but not enough to be perceived as a gel by the rheometer.

As the temperature is increased the time required to reach the smooth region decreases. Theory regarding the gelation mechanism of HE pectin under cold conditions suggests that formation of hydrophobic and hydrogen bonding is separated; i.e. only hydrophobic interactions are initially formed since no acid is present. As GDL is added, acid is slowly released and hydrogen bonding can begin. Thus, with increasing temperature the balance between hydrophobic interactions and hydrogen bonding likely shifts towards the more typical hot gelation mechanism; i.e. they form simultaneously.



Figure 6 shows cold set technology applied to wine gums filled with fish oil / probiotic centres.

The technology has already been commercially launched in the UK with another confectionery product.

CONCLUSIONS

HE pectin has been shown to gel under cold conditions upon acidifier addition. Accurate predictions of onset and gelation are linked to acidifier concentration, pH and temperature; thus allowing processing guides for confectionery products to be made.

Evidence pointing to a new pectin gelation mechanism has emerged. Increasing temperature increases the rate of onset of gelation itself, and is attributed to the balance between hydrophobic interactions and hydrogen bonding. It is

suggested that this balance reverts to the traditional mechanism whereby both types of interaction are formed simultaneously. For reasons of classification any temperature above 40°C is not considered cold setting.

REFERENCES

1. Gilsensan, P. M., Richardson, R.K., and Morris, E.R. (2000) "Thermally reversible acid-induced gelation of low-methoxy pectin." *Carbohydrate Polymers* **41**, 339-349.
2. Lootens, D., Capel, F., Durand, D., Nicolai, T., Boulenguer, P., and Langedorff, V. (2003) "Influence of pH, Ca concentration, temperature and amidation on the gelation of low methoxyl pectin." *Food Hydrocolloids* **17**, 237-244.
3. Vie, R., Azema, N., Quantan, J.C., Touraud, E., and Fouletier, M. (2007) "Study of suspension settling: A approach to determine suspension classification and particle interactions." *Colloid & Surfaces A: Physicochem. Eng. Aspects* **298** 192-200.
4. Mickle, H.C., and Dunstan, D.E. (2002) "Determining the yield stress of xanthan via droplet rise experiments." In *Gums and Stabilisers for the Food Industry 11*, P.A. Williams, and G.O. Phillips (Eds.) Royal Society of Chemistry, Special Publication no.278, pp 120-127.