TECHNICAL APPLICATION
INFORMATION

Herbstreith&Fox

Floating – Scourge of Fruit Processors?

FLOATING –
SCOURGE OF FRUIT PROCESSORS?

Introduction

The use of pectin as gelling and thickening agent in the production of fruit spreads and fruit preparations for industrial processing (e.g. yoghurt fruit preparations) has a long tradition.

In addition to a pleasant texture and a naturally fruit typical flavour the preservation of fruits as well as the even fruit distribution in both glassware and containers are decisive quality criteria in the industrial production of fruit preparations containing whole fruits or fruit pieces (fig. 1).

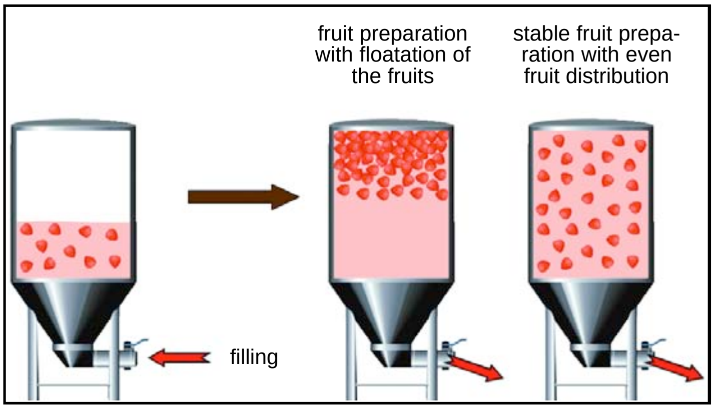

Figure 1: Fruit preparations

In this context, texture and gelling properties of the fruit preparations essentially depend on recipe parameters such as soluble solids content, type of sugar, pH-value, addition of buffer salts, pH-value of the product as well as on the used pectin type and the filling temperature.

In order to prevent floatation, that means the undesired rising of the fruits, the recipe parameters can be modified in a way that, at a defined filling temperature, the gelation process has already started in an extend that the fruits are bound in the emerging gel structure. This effect can result in the so called pre-gelation which often has a negative influence on texture and syneresis behaviour of the final products.

Objective of the tests is to develop a method which is able to provide information if a fruit preparation shows floatation or not. At the same time the influence of measures for prevention of floatation on the texture of the final products is studied.

Beside the sensory visual determination of the floatation behaviour and texture particularly the determination of the rheological parameters yield stress, viscosity and breaking strength are of special importance.

It is objective to show that, at a defined filling temperature, not only a defined viscosity but also the presence of a certain yield stress is necessary to prevent floatation.
Furthermore special pectins are investigated which are able to prevent floatation thus resulting in products with attractive texture.

Physics of floatation

As shown in figure 2 there are 3 forces acting on the particle: Buoyancy F_B, frictional (drag) force F_D and weight force F_W. Both, buoyancy F_B and frictional (drag) force F_D are acting upwards. Buoyancy tends to float the particles and drag force resists the acceleration of gravity. The weight force F_W is the only force acting downwards and results from the gravitational force (Shearer, Hudson, 2008).

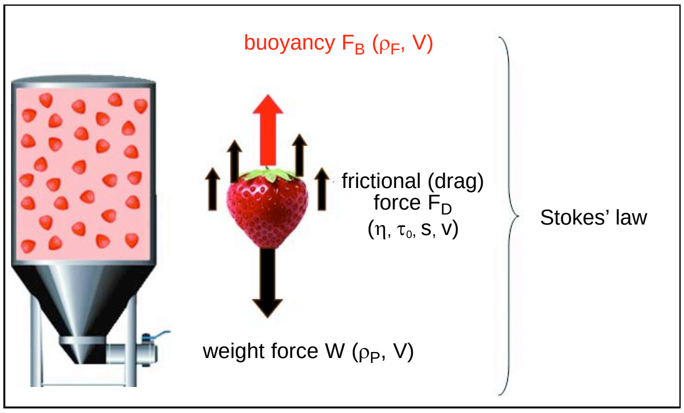

Figure 2: Forces acting on a particle in a fluid

An even distribution of the particles in the fluid is a function of its immobility and results if the described forces are able to counterbalance each other. We obtain:

$$F_W - F_B = F_D \qquad [1]$$

F_W = weight force [N]
F_B = buoyancy [N]
F_D = frictional (drag) force [N]

Acting on the assumption that the particles have a spherical shape and F_D describes the flow resistance of a sphere in an indefinite yield we can replace $F_W - F_B$ by:

$$F_W - F_B = \Delta m \cdot g = V_P \cdot \Delta\varphi \cdot g = V_P \cdot (\varphi_P - \varphi_F) \cdot g \qquad [2]$$

Δm = mass of particle – mass of fluid [kg]
g = gravitational acceleration [m/s²]
V_P = volume of the particle [m³]
$\Delta\varphi$ = density of particle – density of fluid [kg/m³]
φ_P = density of particle [kg/m³]
φ_F = density of fluid [kg/m³]

Using Stokes' law F_D it can be substituted as follows: for $F_D = 3 \cdot \pi \cdot d_P \cdot \eta \cdot v$ where d_P stands for the average diameter of the particle, η for the viscosity of the fluid and v for the particles velocity.

$$F_D = 3 \cdot \pi \cdot d_P \cdot \eta \cdot v \qquad [3]$$

d_P = average diameter of particle [m]
η = viscosity of fluid [Pas]
v = velocity of particle [m/s]

The volume of the particle (assumed as a sphere) can be described as:

$$V_P = \frac{1}{6} \cdot \pi \cdot d_p^3 \qquad [4]$$

Regrouping and rearranging the terms in the above equation we arrive to the following relationship for the particles velocity:

$$v = \frac{d^2 \cdot (\varphi_P - \varphi_F) \cdot g}{18\eta} \qquad [5]$$

If the density of the particle is higher than the fluids density ($\varphi_P > \varphi_F$) the velocity direction is downwards, in contrary if ($\varphi_P < \varphi_F$) the velocity direction is upwards.
Only in the case that both densities are equal ($\varphi_P = \varphi_F$) the particles velocity becomes zero and the particles are immobilized.

Regarding equation [5] the goal of the processor of fruit preparations is to get a velocity as low as possible in order to minimize floatation. Table 1 shows that for this purpose there are different approaches resulting in benefits but also in possible disadvantages:

Measure	**Benefits/Result**	**Possible Disadvantages**
maximize heat viscosity	inrease of η	will destroy fruit pieces
reduce filling temperature	inrease of η	pre-gelation, microbiological problems
adapt density of particles to density of fluid, e.g. increase cooking time	reduction of $\Delta\varphi$	off-taste caused by long cooking-time, long production time
reduce size of fruit pieces	reduction of buoyancy B	fruit pieces too small
maximize agitation	even distribution of fruit pieces	will destroy fruit pieces

Table 1: How to minimize floatation

Regarding equation [5] in the face of viscosity, the particles' velocity can be considered as zero, if the viscosity tends to infinite. This special case occurs if a yield stress τ_0 exists. The yield stress which is needed to prevent floatation is called the critical yield stress $\tau_{0,crit.}$.
Table 2 shows that in practice the necessary critical yield stress must have, depending on the recipe, differing values to prevent reliably floatation of the fruits or fruit pieces. Not until the critical yield stress $\tau_{0,crit.}$ is reached in the production of the fruit preparation, the fruit pieces are immobilized. The critical yield stress depends on the shape of the fruit pieces as well as the soluble solids content of the fruit preparation.

		Tendency to Floatation at Filling Temperature		
Fruit	**Piece Size**	**< 40°Bx**	**40°Bx**	**> 40°Bx**
raspberry	broken	lower ⇦	low	⇨ higher
peach	6mm dices		medium	
cherry	whole fruit		medium	
strawberry	16mm dices		high	
	10mm dices		high	
	whole fruit		very high	

Table 2: Critical yield stress

In order to reach a yield stress in the respective fruit preparations in our tests, the calcium dosage in each recipe was varied. By rheological determination of the yield stress and simultaneous visually observing of the floating behaviour the necessary critical yield stress $t_{0,crit.}$ could be determined for the given filling temperature. Depending on the soluble solids content of the recipe pectins with suitable calcium reactivity were used for the tests.

In order to reach the requested critical yield stress either the calcium dosage or the calcium reactivity of the pectin used can be increased in the given recipe and filling temperature.

Reactivity of low methylester, amidated pectins

Pectin Type		Calcium-reactivity	Setting Rate	Typical Degree of Esterification	Typical Degree of Amidation
Apple Pectin	Citrus Pectin				
Amid AF 005	Amid CF 005	low	slow	35%	15%
Amid AF 010	Amid CF 010	medium	medium	32%	18%
Amid AF 020	Amid CF 020	high	rapid	30%	20%

Table 3: Reactivity of low methylester, amidated pectins

For fruit preparations mainly low methylester, amidated pectins are used. Low methylester, amidated pectins gel with calcium ions according to the egg-box model. Depending on TSS diverse calcium reactivities are necessary.

To find the right pectin for the different TSS ranges in our investigations it is useful to know that low methylester, amidated pectins have different reactivities. The reactivity is a result of different degrees of amidation and esterification.

On the basis of previous tests the pectin with the calcium reactivity suitable for the different soluble solids contents was selected. The most suitable pectins are those which are, due to their reactivity, able to form homogeneously firm set gels covering a wide range and being independent from the calcium dosage.

Figure 3 shows that the most suitable pectin for a very low soluble solids content of e.g. 20% and pH approx. 3.2 is the low methylester, amidated Pectin Amid AF 020 with high calcium reactivity.

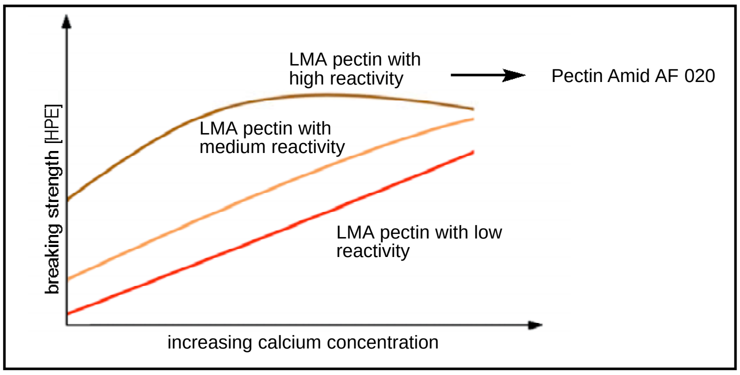

Figure 3: Reactivity of low methylester, amidated pectins (20% TSS, pH 3.2)

As seen in figure 4, the best pectin for a low soluble solids content as e.g. 40% and pH approx. 3.2 is the low methylester, amidated Pectin Amid AF 010 with medium calcium reactivity.

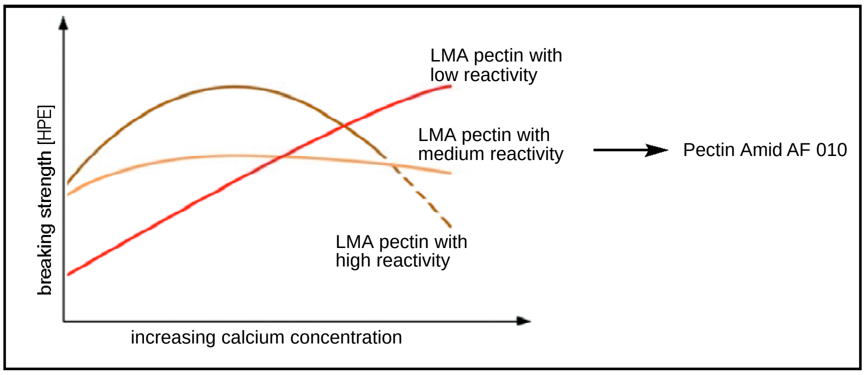

Figure 4: Reactivity of low methylester, amidated pectins (40% TSS, pH 3.2)

For a high soluble solids content as e.g. 60% and pH approx. 3.2 the low methylester, amidated Pectin Amid AF 005 with low calcium reactivity can be used (see figure 5).

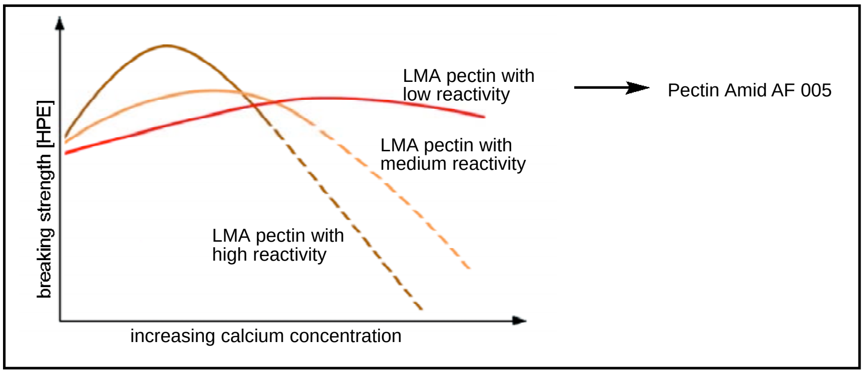

Figure 5: Reactivity of low methylester, amidated pectins (60% TSS, pH 3.2)

Determination of the critical yield stress and viscosity

The determination of the critical yield stress and viscosity at 70°C was done in a cherry fruit preparation which was produced in dependence from the calcium dosage with different soluble solids contents. Prior to determining the yield stress and the viscosity using the rheometer, the fruit preparation was sieved in order to avoid measuring errors caused by fibers and fruit pieces. The yield stress being observed at a calcium dosage with which for the first time there was no floatation of the fruits, is defined as critical yield stress.

The determination of yield stress and viscosity at a specified filling temperature of 70°C was effected using the Rheometer Physica MCR 301. To determine the yield stress the shear stress was reduced logarithmically over time. The assessment can be done e.g. using the tangent method in the log gamma/log tau diagram.

Measuring System:	**Z3 DIN**
shear stress	0.05Pa – 50Pa log ramp, down
time	120s
temperature	70°C

For determining the viscosity the shear rate was increased over the time. The viscosity was determined at a defined shear rate.

Measuring System:	**Z3 DIN**
shear stress	0 – 120s^{-1} linear ramp, up
time	120s
temperature	70°C

Evaluation of floatation

If the fruit preparation was floating at the specified filling temperature has been assessed sensorily (visually) by a sensory research team.

Determination of the setting temperature

For determination of the setting temperature the oscillating rheometer Bohlin CS 10 was used. For the measurement the preparation was placed in the test chamber and then cooled under defined conditions.

Measurement System:	**PP 40 (plate-plate, ⌀ 40mm)**
frequency	1Hz
shear stress	0.06Pa, linear ramp
start temperature	95°C
end-temperature	20°C
cooling rate	2°C/min.
gap	1mm

The setting temperature of the formula was taken as the temperature when, due to the beginning gel formation, the elastic shares increase very strongly resulting in an immensely increased storage modulus G' (tangent method).

Determination of breaking strength

As, due to the fruit pieces contained, it is not possible to determine the breaking strength of cherry fruit preparations, the same products were produced using strawberry pulp. The determination of breaking strength (20°C) was done with the Herbstreith Pektinometer Mark IV.

Table 4 illustrates again the recipe parameters used as well as the methods.

Material	**Methods**
production of cherry fruit preparations with maintained cherries • increase of Ca^{2+} dosage • variation of TSS (30 – 60%) • T_{fill} = 70°C	• sensory evaluation of floatation at T_{fill} and texture of the final product → sensory research team
	• rheological determination of yield stress and viscosity at T_{fill} (70°C) • rheological determination of setting temperature
production of strawberry fruit preparations with strawberry pulp • increase of Ca^{2+} dosage • variation of TSS (30 – 60%) • T_{fill} = 70°C	• rheological determination of yield stress and viscosity at T_{fill} (70°C) • rheological determination of breaking strength (20°C)

Table 4: Material and Methods

Results

For each tested soluble solids content (30 – 60%) it could be seen that the typical curve flow of yield stress and viscosity recur in dependence from calcium dosage.

Fig. 6 shows such a typical curve flow. Regarding the yield stress curve in dependence from calcium dosage, three ranges with different increase can be seen. If a tangent is applied to the curve in each range, two intersections result (A and B). A yield stress is formed as soon as a gel net in the gel preparation is formed. The more calcium ions available, the more bonding points and the higher the yield stress determined (range 1).

Starting at a defined calcium dosage (point A) the gel formation starts already at a temperature above the measuring temperature. This can be shown with the strawberry fruit preparation as the breaking strength curve has its maximum exactly in point A (see figure 8). If the maximum breaking strength exceeds due to a higher calcium dosage, the setting temperature is above the filling temperature and the gels become again weaker due to pre-gelation. Pre-gelation means that an over-reaction between the pectin molecules and the calcium ions occurs. Fine gel particles are formed, the gel arrangement looses its elastic character and the texture becomes pasty resulting in a reduction of gel strength. At mechanical treatment the gel looses water, syneresis occurs.

The velocity of gel formation gets higher the more calcium ions are available (decrease of setting time). By overlapping with this gelling process the yield stress in range 2 between A and B increases intensely.

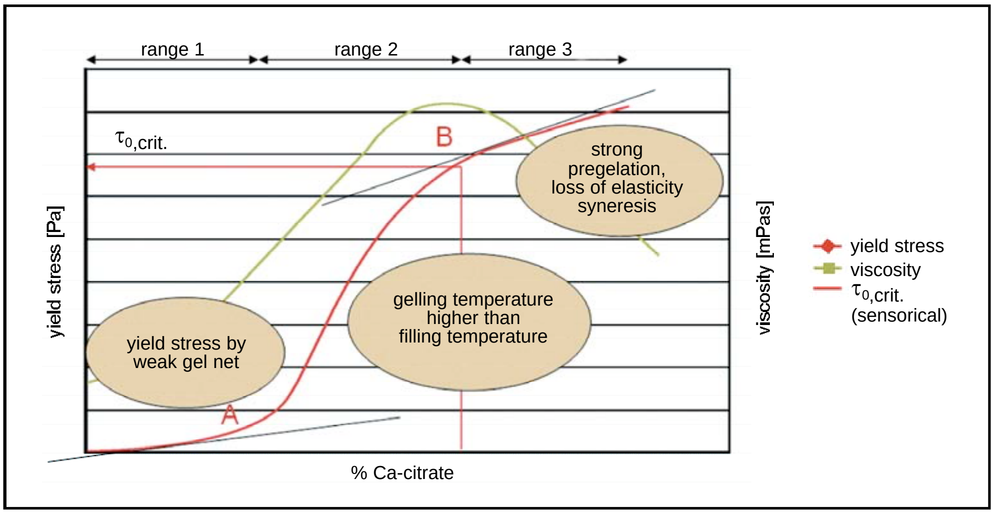

Figure 6: Critical yield stress at 70°C

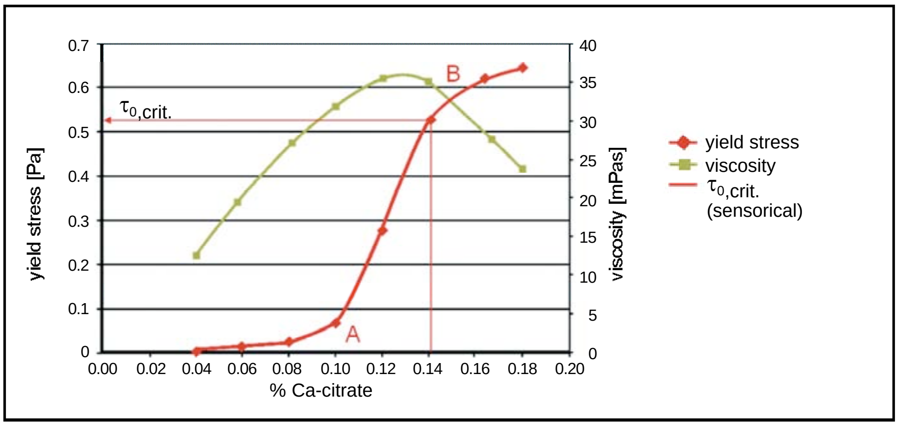

Figure 7: Yield stress and viscosity of cherry fruit preparations (70°C), 30% TSS, pH 3.2, Pectin Amid AF 020

If the calcium dosage will be still increased, the products with calcium dosages above B are pre-gelled to an extent that the gel looses water already when reaching the measuring temperature (70°C). This results in a strong decrease of both viscosity and also the elastic shares of the fruit preparation. Thus the yield stress is no longer able to inrease intensely.

In all tests the critical yield point (yield point, at which for the first time there was no floating) was reached at higher calcium dosages than in point A and often in the range of maximum viscosity.
It is conspicuous that the critical yield stress determined lies in the range of calcium dosages above A and often in the range of maximum viscosity. In practice this means that with the addition of calcium ions the critical yield stress can be reached, the texture of the resulting fruit preparation, however, is pre-gelled with high tendency to synersis.

Figure 7 shows viscosity and yield stress of the sieved cherry fruit preparations in dependence from the calcium dosage.
The curve of the yield stress shows a typical s-shaped flow in dependence from the calcium dosage and the viscosity of the tested fruit preparations increases up to reaching a maximum and then decreases again having reached a defined calcium concentration. By means of sensory assessment of the floatation behaviour a critical yield stress of 0.53Pa could be determined which is in the range of the viscosity maximum when calcium is added.

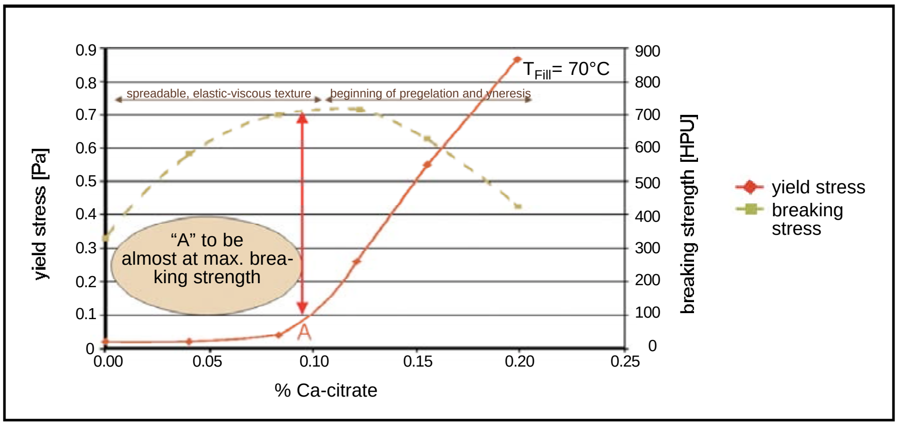

Figure 8: Yield stress (70°C) and breaking strength (20°C) of strawberry fruit preparations, 30% TSS, pH 3.2, Pectin Amid AF 020

Figure 8 shows breaking strength and yield stress of the fruit preparations produced with strawberry pulp in dependence from the calcium dosage. In dependence from calcium dosage, the curve of yield stress displays a flow comparable to that of the cherry fruit preparations. The breaking strength increases up to reaching a defined calcium dosage and then decreases again. The texture of the fruit preparation is spreadable and gets a slightly elastic-viscous character with increasing calcium dosage. With calcium dosages above the maximum value of the breaking strength the products become softer and show an increasingly pre-gelled texture.

Figures 9, 11 and 13 illustrate the viscosity and yield stress of cherry fruit preparations in dependence from calcium dosage at 40, 50 and 60% TSS. The fruit preparations have been manufactured using the pectin appropriate for this TSS range. All TSS ranges investigated show comparable curve flows of yield stress and also viscosity. The higher the critical yield stress determined, the higher the soluble solids content of the fruit preparation. With increasing soluble solids content the viscosity of the fruit preparation increases implicating the necessary critical yield stress moving closer to A.

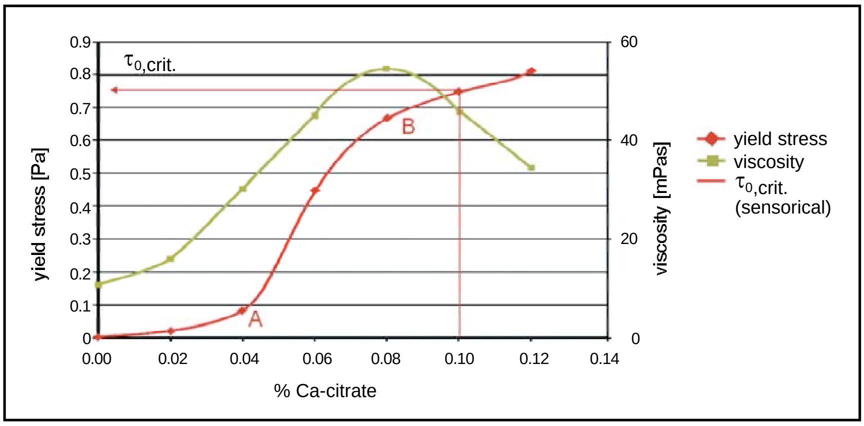

Figure 9: Yield stress and viscosity of cherry fruit preparations (70°C), 40% TSS, pH 3.2, Pectin Amid AF 010

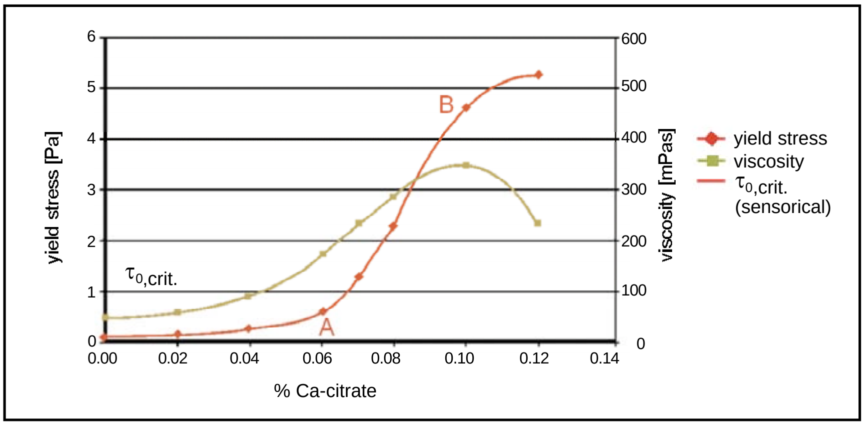

Figure 11: Yield stress and viscosity at 70°C of cherry fruit preparations, 50% TSS, pH 3.2, Pectin Amid AF 005

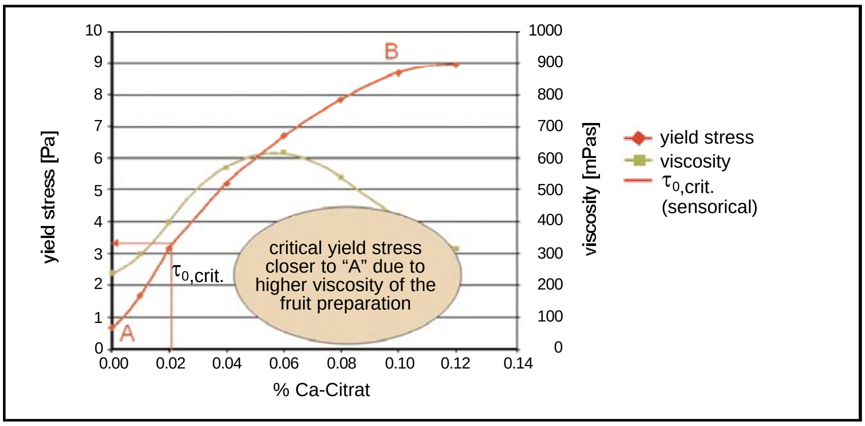

Figure 13: Yield stress and viscosity of cherry fruit preparations (70°C), 60% TSS, pH 3.2, Pectin Amid AF 005

Figures 10, 12 and 14 show yield stress and breaking strength of strawberry fruit preparations in dependence from calcium dosage at 40, 50 and 60% TSS. For the TSS range the appropriate pectin was used.

The maximum breaking strength of all investigated fruit preparations is reached in A, at higher calcium dosages pre-gelation occurs resulting in a loss of gel strength.

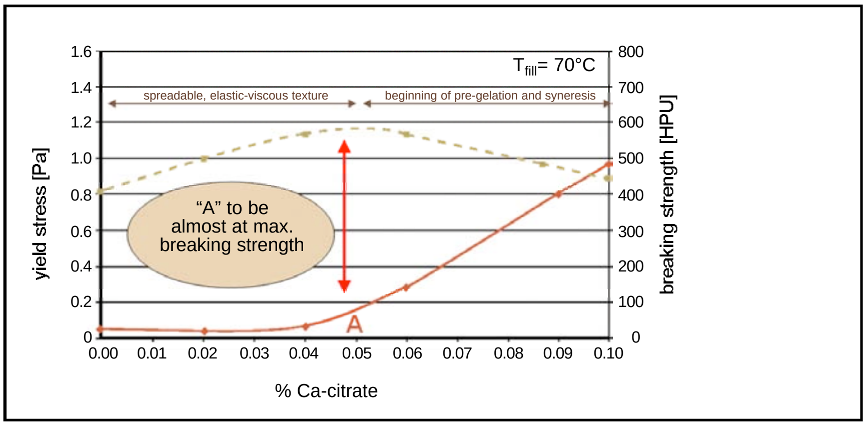

Figure 10: Yield stress (70°C) and breaking strength (20°C) of strawberry fruit preparations, 40% TSS, pH 3.2, Pectin Amid AF 010

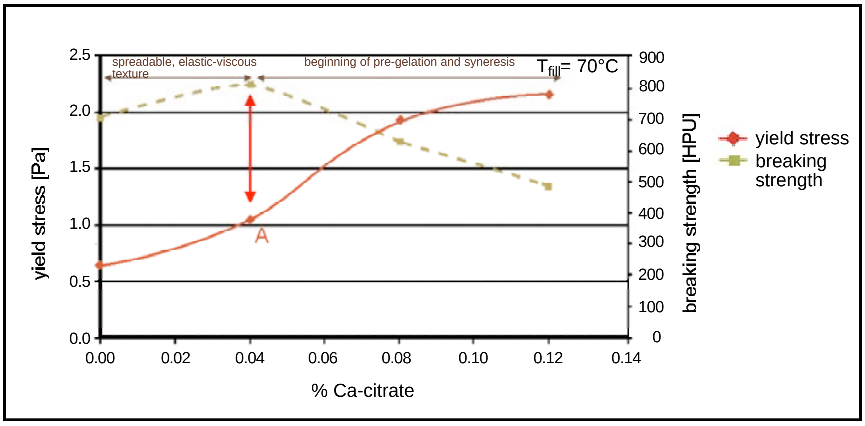

Figure 12: Yield stress (70°C) and breaking strength (20°) of strawberry fruit preparations, 50% TSS, pH 3.2, Pectin Amid AF 005

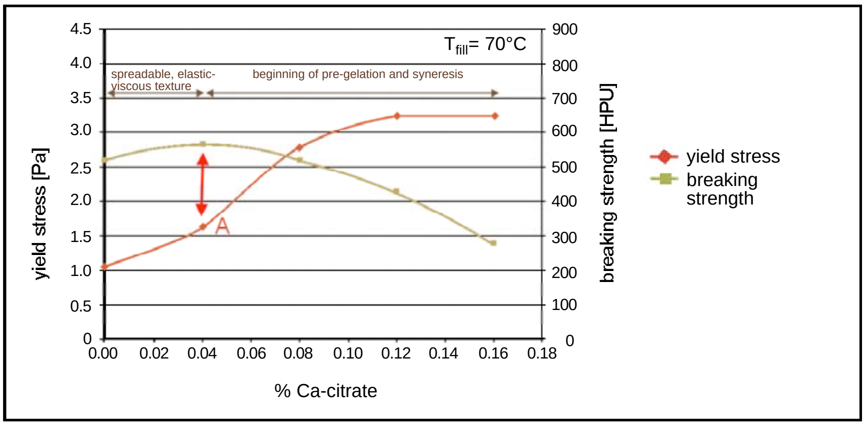

Figure 14: Yield stress (70°C) and breaking strength (20°C) of strawberry fruit preparations, 60% TSS, pH 3.2, Pectin Amid AF 005

As a summary, figure 15 illustrates the yield stress of cherry fruit preparations with different soluble solids contents in dependence from the calcium dosage, also the sensorily determined critical yield stress for each soluble solids content is shown.

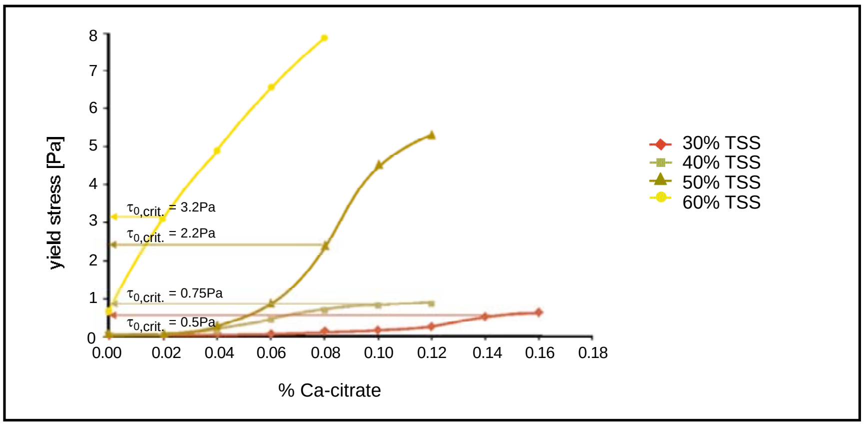

Figure 15: Yield stress and viscosity of cherry fruit preparations (70°C), 30 – 60% TSS, pH 3.2

Figure 16 shows the critical yield stress in dependence from soluble solids content for the tested cherry fruit preparations (filling temperature 70°C). It becomes obvious that with increasing soluble solids content the necessary critical yield stress has to be higher to prevent the fruits from floating. It has to be considered that these values cannot be seen as absolute values, they are valid only for the present conditions.

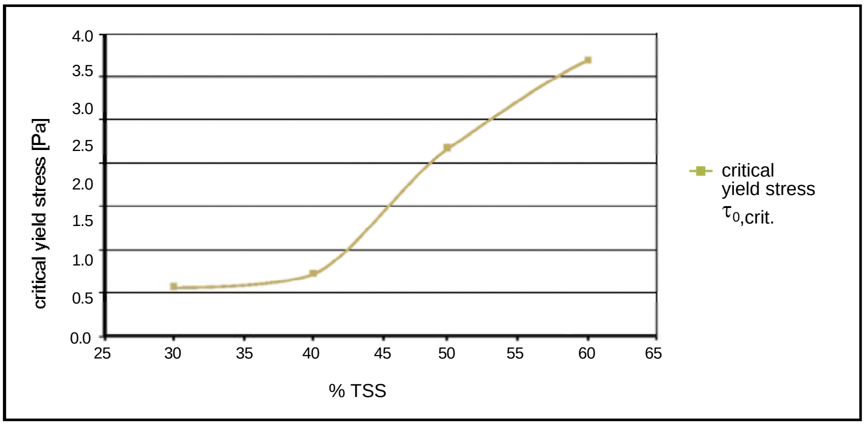

Figure 16: Critical yield stress of cherry fruit preparations (70°C), 30 – 60% TSS

Special pectins to prevent floatation

Floatation can only be surely prevented by presence of a yield stress which can be reached by altering the recipe parameters (in this case the calcium dosage), the resulting products, however, do not meet the requirements of producers of fruit preparations regarding texture.

H&F has developed special pectins which are, due to their manufacturing process, able to form a yield stress lying above the critical yield stress already without separate calcium addition.

Furthermore the products are characterized by a spreadable up to elastic-viscous texture. Figure 17 shows yield stress and breaking strength of strawberry fruit preparations with a soluble solids content of 40% manufactured with pectins A and B. The critical yield stress determined with pectin A (Pectin Amid AF 010) in this system is approx. 0.75Pa and lies in B. The fruit preparation being manufactured with the special pectin B shows, already without any addition of calcium, a yield stress which lies with 1.1Pa clearly above the necessary critical yield stress. The maximum of the breaking strength curve of the fruit preparations manufactured with Pectin B is reached at a calcium dosage of approx. 0.05%. At calcium dosages below this maximum the resulting products have an elastic-viscous and spreadable texture without pre-gelation.

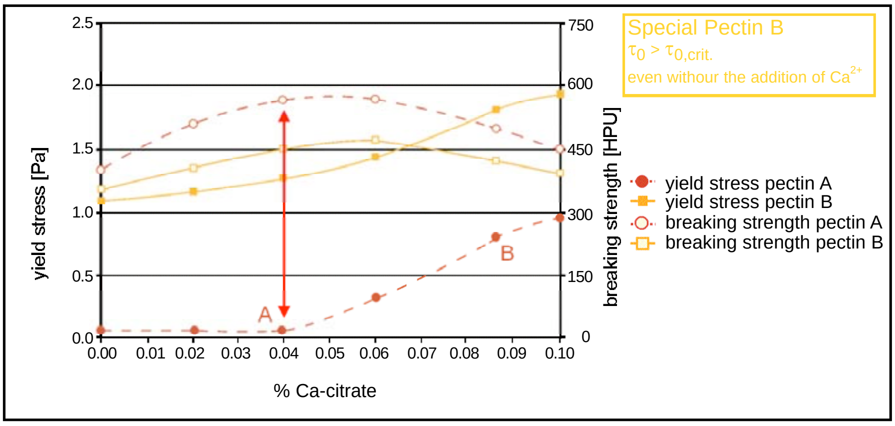

Figure 17: Yield stress (70°C) and breaking strength (20°C) of strawberry fruit preparations, 40% TSS, pH 3.2

Thus with the special pectins of H&F fruit preparations can be produced for which floatation of the fruits is surely prevented over a wide soluble solids range and at the same time – depending on the recipe – spreadable-viscous up to elastic-viscous texture without pre-gelation and syneresis are formed.

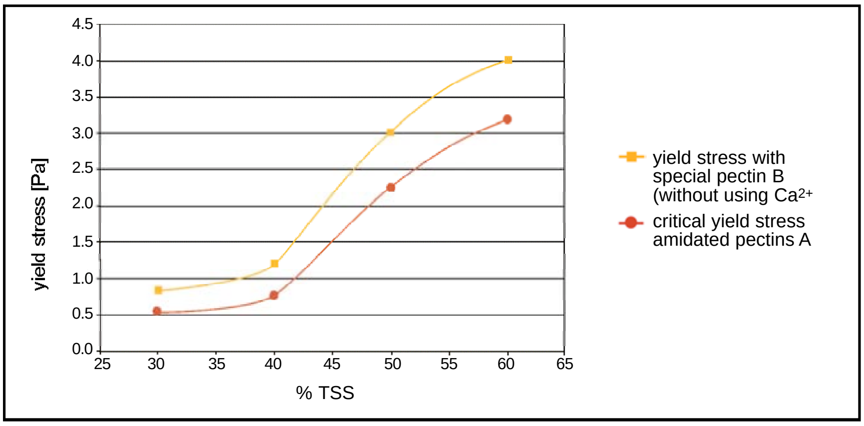

Figure 18: Critical yield stress and yield stress of fruit preparations made with special pectins in dependence from TSS (cherry fruit preparation pH 3.2, 70°C)

Figure 18 illustrates the critical yield stress and the yield stress, which is reached with H&F special pectins without a separate calcium addition, in dependence from soluble solids content. The values apply for the tested cherry fruit preparation and a filling temperature of 70°C. It is obvious that H&F special pectins always reach a yield stress which is higher than the critical yield stress thus preventing floatation reliably.

With determining the elastic and viscous shares it can be shown that H&F special pectins for floatation prevention have mainly elastic shares already at very high temperatures, but the gelation process did not start yet. Measuring conditions correspond to the determination of the setting temperature of a jelly fruit mass using the Rheometer Bohlin CS 10 (oscillating measurement: temperature gradient at constant frequency and amplitude). Measuring curve G' and G" resp. the phase shift angle δ provide information on the ratio of elastic and viscous shares. If the elastic shares of a gel preparation overbalance, then a yield stress is available.

Figure 19 shows that H&F special pectins are able to form a yield stress over a wide temperature range and already at very high temperatures without the gelling process having started yet.

Due to the yield stress, fruit preparations manufactured with Pectin B have more elastic shares (G' > G") already at high temperatures, the phase shift angle is δ < 45°. Fruit preparations without yield stress have more viscous shares (G" > G'), the phase shift angle δ is higher than 45°.
The real gelation, however, does not start until reaching the temperature at which the elastic shares increase disproportionately high. After assessing the G' curve using the tangent method, for both pectins A and B 65°C are determined.

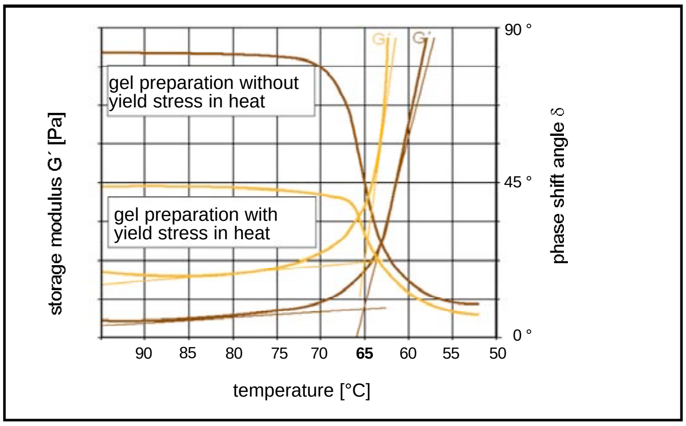

Figure 19: Determination of setting temperature

Conclusions

For the prevention of floatation fruit preparations have to have a defined yield stress (critical yield stress $t_{0,crit.}$) when reaching the filling temperature. If this yield stress is reached by altering the recipe parameters, e.g. by increasing the calcium dosage, indeed products with even fruit distribution are obtained, however the texture is pre-gelled and the product tends to syneresis.

With specially developed pectins from H&F it is possible to manufacture fruit preparations which form a yield stress already during the heating process and without separate addition of calcium. With that the floating of fruits and fruit pieces in fruit preparations is reliably prevented. The products are characterized by their attractive texture and are also excellently suited for industrial processing.

Further information on the anti-floating pectins of H&F you can find in our new technical application information “ Pectins with Anti-Floating Effect”.

References:

Shearer, Scott A., Hudson, Jeremy R., Fluid Mechanics: Stokes’ Law and Viscosity. Measurement Laboratory. Investigation No. 3. http://www.engr.uky.edu/~egr101/ml/ML3.pdf

Research and Development
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