

# Rheological methods for characterization of industrially produced jam

Marie Skov Pedersen<sup>1,3</sup>, Kristian Hovde Liland<sup>2</sup>, Tove Devold<sup>1</sup>, Elling-Olav Rukke<sup>1</sup>, Elin Mittet<sup>3</sup>, and Reidar Barfod Schüller<sup>1</sup>

<sup>1</sup>Norwegian University of Life Sciences, Faculty of Chemistry, Biotechnology and Food Science, P.O. Box 5003, N-1433 Ås, Norway

<sup>2</sup>Norwegian University of Life Sciences, Faculty of Science and Technology, P.O. Box 5003, N-1433 Ås, Norway

<sup>3</sup>Orkla, P.O. Box 423 Skøyen, N-0213 Oslo, Norway

## ABSTRACT

The food industry requires quick and reliable methods for characterization of properties of industrially produced jams. It was of interest to investigate if it was possible to detect significant differences when the type of pectin in the recipe was changed.

Three types of jams have in this study been characterized using several different methods, both using a texture analyser and a rheometer. The method used in the texture analyser was a classical TPA (Texture Profile Analysis)<sup>1, 2</sup> with post-processing using a suitable macro.

The results show that rheological methods reveal many of the important properties of industrial jams. Some of these properties can be linked to sensory perception.

## INTRODUCTION

The goal of this work was to investigate which measurement methods that are suitable to characterize the texture and rheological properties of industrial jam. It was also of interest to be able to detect the difference of different pectins or pectin mixtures in the recipes.

The texture in jam depends on several factors like the concentration of berries, sugar and additives<sup>3</sup>. Concentration of pectin and sugar affect the strength of the jam, which is investigated in this report. There are many different pectins on the market and the

type of pectin is often chosen to achieve desired product properties and the required texture. A low concentration of pectin may not give the desired texture by not making a gel, while a too high concentration can cause production problems and a non-cohesive jam<sup>4</sup>.

The attributes from a parallel sensory study were also made available to increase the number of product properties included in the study. A part of the study was trying to determine which variables are correlated.

## MATERIALS AND METHODS

### The jams

Several different jams were produced using different types of fruits (strawberry, raspberry and orange) to conserve and make good quality products<sup>3, 5, 6</sup>. The recipes used different types of pectin<sup>4, 7, 8</sup> as shown in Table 1. The base recipe type is indicated by capital letters and the pectin types used by identifying numbers. The experimental design thus made it possible to assess if the use of different types of pectin or pectin mixtures could be observed by applying several rheological methods.

Pectins are complex anionic polysaccharides. For their use as a food additive, they are classified either as HM (high methoxy) or LM (low methoxy) types based on the percent of methylated carboxyl groups of the galacturonic acid units<sup>3, 4, 8, 9</sup> described as the degree of methyl-

esterification (DM). All the pectins in this study were LM pectins, with a DM from 24 – 35. Some of the pectins used were amidated with degree of amidation (DA) ranging from 13 to 23<sup>4</sup>. Their calcium reactivity differed from low to high.

The jams were produced by first adding fruit, water and an antifoaming agent during heating until boiling before adding the sugar. The pectin solution was made by blending pectin and potassium sorbate with hot water and mixing with an immersion blender until the pectin was completely dissolved. The pectin solution was then added to the fruit and sugar blend and was boiled further for 5 minutes. When finished boiling, the acid was added, and the evaporated water was compensated for by adding water. Thereafter the jam was cooled until filling temperature and one sample was filtrated through a colander layered with a chiffon-cloth to get a sample of the continuous phase. After filling, the glasses (0.5 l jam and 0.4 l filtrate) were placed in room temperature for three to four days before moving.

The jam was used in the BMS and for the sensory evaluation, while the filtrate was used for the plate/plate measuring system. The TPA was used on both.

Table 1: Test sample description

| Name             | Recipe | Pectin type |
|------------------|--------|-------------|
| 60 Raspberry 1   | A      | 1 and 6     |
| 60 Raspberry 2   | A      | 10          |
| 40 Raspberry 1   | B      | 2 and 4     |
| 40 Raspberry 1   | B      | 8           |
| Strawberry 1     | C      | 1 and 6     |
| Strawberry 2     | C      | 10          |
| NSA Strawberry 1 | D      | 1 and 2     |
| NSA Strawberry 2 | D      | 10          |
| Orange 1         | E      | 1 and 6     |
| Orange 2         | E      | 8 and 9     |
| Orange 3         | E      | 8           |

60 and 40 indicate vol% berries.  
NSA denotes no sugar added.

### Sensory evaluation

During the sensory evaluation 8 semi-trained panelists were asked to identify the intensity of hardness, elasticity, and coating in mouth on a scale from 1 – 5, where 1 was low/little and 5 high/much. Hardness and elasticity were determined using a spoon, while the coating was analyzed by how much the jam coated the mouth when eating. The results were used to investigate correlations in jam.

### Texture analyser setup

A texture profile analysis (TPA)<sup>1, 2</sup> was performed using a texture profiler from Stable Micro Systems Ltd., UK, using data collected from a normal two-bite test procedure. A Delrin 10 mm diameter probe was used, and this was set to penetrate 5 mm into the jam. The test speed during measurements was 2 mm/s.

The standard TPA macro, supplied by the manufacturer, was used to analyse the data, and several variables were determined:

- Hardness
- Fracturability
- Adhesiveness
- Springiness
- Cohesiveness
- Gumminess
- Chewiness
- Resilience

### Rheometer setup

An MCR301 (Anton Paar) rheometer was used in these tests. Several measuring systems were used; a BM 12/72.5 ball measuring system as shown in Fig. 1 and a normal PP50 plate/plate measuring system.

The ball measuring system was used to determine the viscosity at different shear rates during a full revolution for jams containing fruits or berries.

The plate measuring system was used for all the measurements made on the filtrate, amplitude sweeps, viscosity, and structure build-up.



Figure 1. BM 12/72.5 measuring system with an eccentrically positioned 12 mm diameter stainless steel ball.

### Statistical analysis

Significant differences between the samples were determined using ANOVA (ANalysis Of VAriance) with the statistical software package R<sup>10</sup>.

Correlation matrices were calculated using the statistical software package R.

### Structure build-up modelling

After the destructive rotational viscosity measurements, the rheometer was set to operate in oscillation mode in the LVE range monitoring how  $G'$  increased with time<sup>11-14</sup>. An example of this structure build-up is shown in Fig. 2 where  $G'$  clearly increases with time.

The growth data was curve fitted in MATLAB<sup>15</sup> to Eq. 1., determining the best values of the constants  $A, B, C$  by the MATLAB-function *nlinfit*. The derivative, the growth rate at any time,  $t$ , can be calculated using Eq. 2.

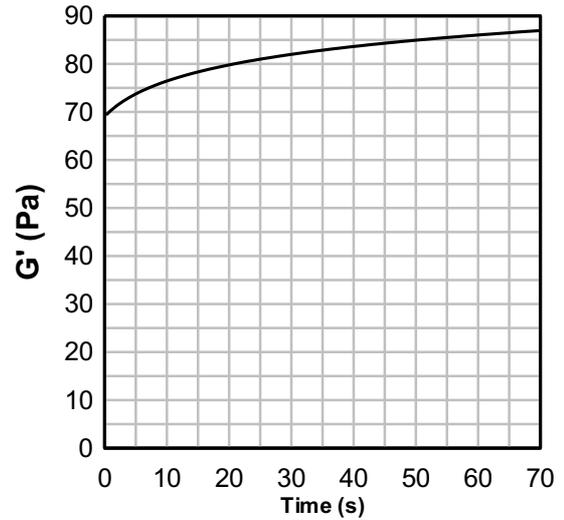


Figure 2. Structure build-up of Strawberry 1 type jam.

$$G' = A \ln(t - B) + C \quad (1)$$

$$\frac{dG'}{dt} = \frac{A}{t - B} \quad (2)$$

Values of the structure build-up rate can be determined from Eq. 2, and  $t = 30$ s was chosen for making the comparisons. The magnitude of the structure build-up over a time interval is expressed by Eq. 3:

$$\Delta G'_{1-2} = G'_{t_2} - G'_{t_1} = A \ln \frac{t_2 - B}{t_1 - B} \quad (3)$$

where a one-minute time interval is chosen to be from time 10 s to time 70 s. The variables  $A, B$  and  $C$  are shown in Fig. 7.

### Correlation coefficients between variables

All the measurements from each recipe were merged as datasets, and correlation matrices between the variables were generated using R to detect variables that exhibit strong positive or strong negative correlation.

All measurements were made at the same temperature.

## RESULTS

### Texture analyser results

The texture analyser with the TPA macro generated results for many variables. Hardness values for the different jams is shown in Fig. 3. Variables are pre-labelled TAF for the filtrates and TAJ for the jams containing berries or fruit in Fig. 7.

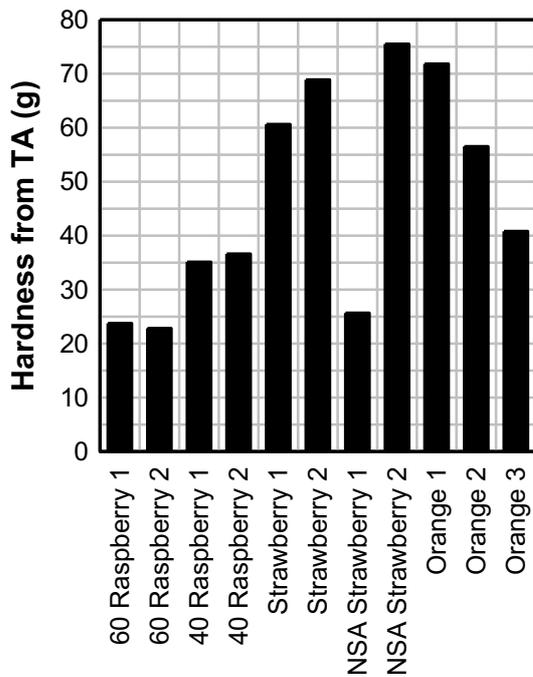


Figure 3. Hardness from TPA measurements.

### Rheometer results

The ball measuring system successfully measures the viscosity at a range of shear rates. The jams and the filtrates were all shear thinning. Thus, two viscosities and a slope are given for both the ball system results and the plate system results. The first viscosity is at low shear rate, the second viscosity is at the high shear rate, and the slope is descriptive of the downward sloping line between the two points.

The amplitude sweep data, and the LVE macro in RheoPlus, successfully determined the stiffness, the strength, and the strain limit of all the filtrate samples at a 3% reduction in  $G'$ . The strain limit results are shown in Fig.

4, the strength results in Fig. 5 and the stiffness results in Fig. 6.

The viscosity of the filtrates was measured in rotation at a range of different shear rates.

The rate of texture build-up differs between the jams, but the magnitude of the increase in  $G'$  is essentially the same.

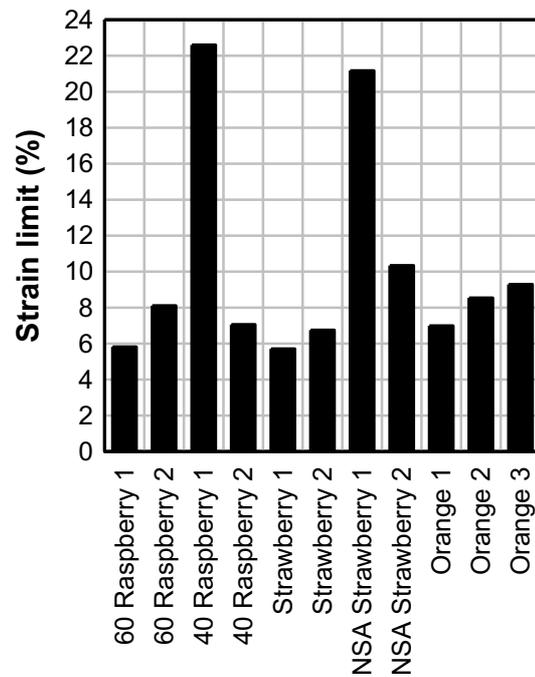


Figure 4. Strain limit values from amplitude sweep (AS) measurements.

### Sensory variables

Three variables were determined by a sensory panel. These were a hardness, an elasticity, and a variable denoted coating being a measure of how well the jam managed to coat the surfaces in the mouth.

### Correlation coefficients

A typical plot of correlation coefficients is shown in Fig. 7. The colour black indicates a coefficient of +1, while white indicates -1. The size of the marker is also proportional to the magnitude of the coefficient.

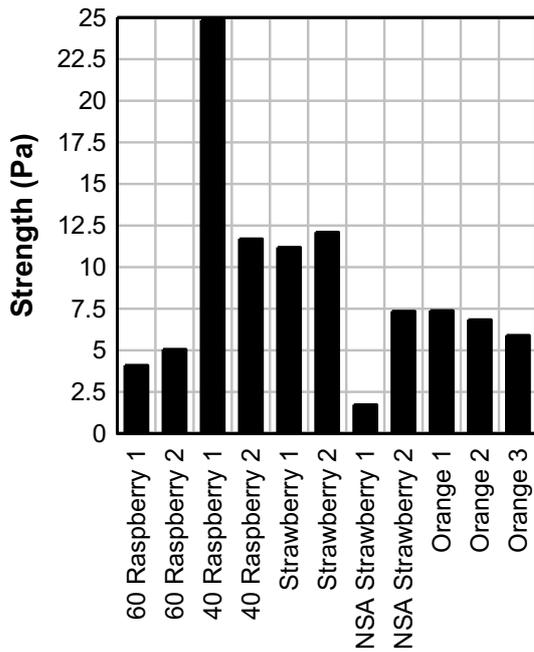


Figure 5. Strength values from AS measurements.

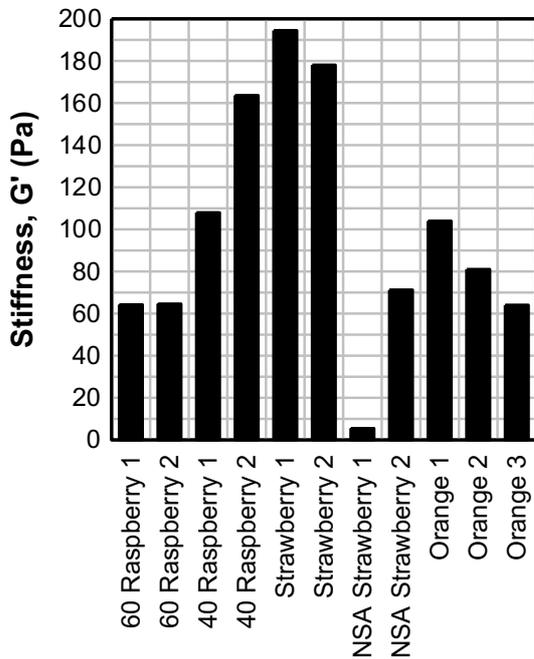


Figure 6. Stiffness values from AS measurements.

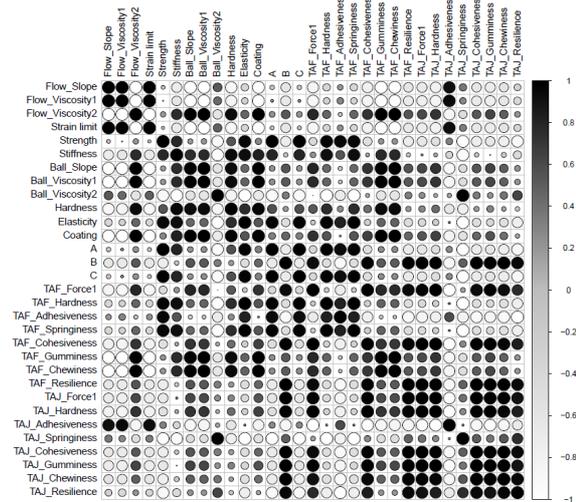


Figure 7. Correlation matrix for 'Orange 1' type jam. The values vary from -1 to +1. The size of the marker increases with magnitude, and the grey scale on the right indicates numerical value; black indicates +1 and white indicates -1.

## DISCUSSION

Significant differences were observed for all the variables, except fracturability, as this property was only observed in very few samples from the TPA. There was not found any or very few differences between the jams of '60 Raspberry' and the strawberry jams for the different parameters, however there were many differences between '40 Raspberry' and 'Orange'. One of the samples made from strawberry, 'NSA Strawberry 1', did not get the expected texture and did not make a typical gel that was expected. This may explain all the differences between these two jams in all the analysis and variables. The differences found between the jams can be explained by differences in the pectin-types, combinations and concentration, where their degree of methylation and amidation were different<sup>4, 9</sup>. However, the TPA was able to detect differences for some of the jams for all variables.

A similar pattern of significant differences between the jams were found in the viscosity analysis. The filtrates showed differences in stiffness, strength, and the

strain limit from the amplitude sweep measurements. It seems like the variables showed largest differences between the '40 Raspberry' and the orange jam. There were also found large differences between each kind of jam, which indicates that the analysis can detect differences in the jam and filtrate.

We observe that there were strong positive correlations between many of the variables, and strong negative correlations between others. There are also variables that seem to have essentially no correlation, shown by the presence of small diameter grey colour symbols in Fig. 7.

It was expected to find some correlation patterns that were similar for all the jams. However, this was not seen, and no general conclusions on the interaction between variables could be drawn. If there had been more samples and several more repetitions of the different analyses, the data may have revealed more information and clearer relationships.

## CONCLUSIONS

The conclusions from this study can be summarized as follows.

- The use of different types of pectin in the jam recipe was for some of the recipes detected by significant differences in the following variables:
    - Hardness
    - Elasticity
    - Coating
    - Adhesion
    - Springiness
    - Cohesion
    - Gumminess
    - Chewiness
    - Resilience
  - The structure build-up differed between the recipes, especially with respect to the rate of structure build-up.
- Some of the rheological variables could be linked to sensory variables as seen in the correlation matrices.
  - The correlation matrices clearly showed the relationships between the variables, but it was not possible to draw any general conclusions on the interaction between variables.

## ACKNOWLEDGEMENT

The authors would like to thank Orkla Foods Norway for the contribution and support to the project and allowing publication of these results.

## REFERENCES

1. Nishinari, K., K. Kohyama, H. Kumagai, T. Funami, and M.C. Bourne, (2013), "Parameters of texture profile analysis", *Food Science Technology Research*, **19**(3): p. 519-521.
2. Rahman, M.S. and S.A. Al-Farsi, (2005), "Instrumental texture profile analysis (TPA) of date flesh as a function of moisture content", *Journal of Food Engineering*, **66**(4): p. 505-511.
3. Vibhakara, H.S. and A.S. Bawa, *Manufacturing Jams and Jellies*. 2007, Blackwell Publishing: Ames. p. 187-204. 9780813808949.
4. May, C.D., (1990), "Industrial pectins: Sources, production and applications", *Carbohydrate Polymers*, **12**(1): p. 79-99.
5. Alves, L.R., J.R. Battochio, J.M. Cardoso, L.L.M.L. De Melo, and V.S. Da Silva, (2008), "Time-Intensity profile and internal preference mapping of strawberry jam", *Journal of Sensory Studies*, **23**(1): p. 125-135.
6. Patras, A., N.P. Brunton, B.K. Tiwari, and F. Butler, (2011), "Stability and Degradation Kinetics of Bioactive Compounds and

Colour in Strawberry Jam during Storage", *Food and Bioprocess Technology*, **4**(7): p. 1245-1252.

7. Willats, W.G.T., C. Orfila, G. Limberg, H.C. Buchholt, G.J.W.M. Van Alebeek, A.G.J. Voragen, S.E. Marcus, T.M.I.E. Christensen, J.D. Mikkelsen, B.S. Murray, and J.P. Knox, (2001), "Modulation of the degree and pattern of methyl-esterification of pectic homogalacturonan in plant cell walls: Implications for pectin methyl esterase action, matrix properties, and cell adhesion", *Journal of Biological Chemistry*, **276**(22): p. 19404-19413.

8. Willats, W.G.T., J.P. Knox, and J.D. Mikkelsen, (2006), "Pectin: New insights into an old polymer are starting to gel", *Trends in Food Science and Technology*, **17**(3): p. 97-104.

9. Löfgren, C., S. Guillotin, and A.M. Hermansson, (2006), "Microstructure and kinetic rheological behavior of amidated and nonamidated LM pectin gels", *Biomacromolecules*, **7**(1): p. 114-121.

10. *R Core Team (2020). R: A language and environment for statistical computing.* 2020, R Foundation for Statistical Computing, Vienna, Austria.

11. Alonso-Mougán, M., F. Mejjide, A. Jover, E. Rodríguez-Núñez, and J. Vázquez-Tato, (2002), "Rheological behaviour of an amide pectin", *Journal of Food Engineering*, **55**(2): p. 123-129.

12. Chan, S.Y., W.S. Choo, D.J. Young, and X.J. Loh, (2016), "Thixotropic supramolecular pectin-poly(ethylene glycol) methacrylate (PEGMA) hydrogels", *Polymers*, **8**(11).

13. Chan, S.Y., W.S. Choo, D.J. Young, and X.J. Loh, *Pectin as a rheology modifier:*

*Origin, structure, commercial production and rheology.* 2017, Elsevier Ltd. p. 118-139.

14. Lootens, D., F. Capel, D. Durand, T. Nicolai, P. Boulenger, and V. Langendorff, (2003), "Influence of pH, Ca concentration, temperature and amidation on the gelation of low methoxyl pectin", *Food Hydrocolloids*, **17**(3): p. 237-244.

15. *MATLAB R2019a.* 2019, Natick, Massachusetts: The MathWorks Inc.