# FUNCTIONALISATION OF PECTIN BY ULTRA HIGH PRESSURE HOMOGENISATION

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# INTRODUCTION

PECTIN:

- Well-known plant-based hydrocolloid
- Found numerous applications in food and other related industries
- Usually commercially obtained from two major sources, apple pomace and citrus peel
- Versatile functionalities predetermined by its structure
- Different functionalities are obtained by modified structure (e.g. enzymatically, chemically etc.)

# INTRODUCTION

ULTRA HIGH PRESSURE HOMOGENISATION (UHPH):

- Mechanical technique
- Known for its ability to modulate the structure and functionality of polysaccharides and different polymers
- Based on forcing a dispersion to flow at a high velocity through a narrow passage, resulting in turbulence, high shear, cavitation, and temperature increase

# AIM OF THE STUDY

The aim of this work was to explore possibility to alter structure of pectin by pilot-scale ultra-high dynamic pressure treatment, as a physical modification approach, in relation to the impact on gel properties.

# MATERIAL & METHODS

1. MATERIAL
2. SAMPLE PREPARATION AND HPH TREATMENT
3. PECTIN CHARACTERISATION

    1. Molecular weight, dispersity and intrinsic viscosity measurements
    2. Apparent viscosity
    3. Gel strenght
    4. Gelation speed and melting point

# 1. MATERIAL

- Citrus unstandardized high esterified pectins (Pektin Classic CU-L 033/20 provided by Herbstreith & Fox GmbH & Co.KG – **CU pectin**
- Apple unstandardized high esterified pectins (Pektin Classic AU-L034/20), provided by Herbstreith & Fox GmbH & Co.KG – **AU pectin**.
- Pectin powders had recorded moisture level of ~10%.

# 2. SAMPLE PREPARATION AND UHPH TREATMENT

PECTIN SOLUTION PREPARATION:

- 2.5% (m/m) CU and AU pectin solutions prepared.
- Pectin powder dispersed in water (~80.0 °C) and mixed for 5 minutes → cooled to ~20 °C with plate heat exchanger (→ adjusted from pH 3 to 6 with NaOH for molecular characterization).

UHPH TREATMENT:

- Pilot-scale, two-stage ultra high pressure homogenizer (*Modell FPG 11300:350, Stansted Fluid Power Ltd., Essex, UK).*
- ***First stage treatment*** at 3 pressure levels: 50, 150 and 250 MPa; ***Second stage treatment*** 8 MPa.

GEL PREPARATION:

- Pectin gels prepared from pectin solutions, according to adapted IPPA °SAG method
- Gel concentration 2.25% (m/m).

# 3. PECTIN CHARACTERIZATION

## MOLECULAR WEIGHT, DISPERSITY AND INTRINSIC VISCOSITY MEASUREMENTS

- Characterization of the UHPH treated pectins was done based on Chirug et al. (2018) developed method.

## APPARENT VISCOSITY

- Measurements of the flow properties were performed according to the guidelines of the German Institute of Standardisation Norm (DIN 53019)
- Cone-plate (cone-Ø 6 cm) rotary rheometer (AR 2000, TA Instruments, New Castle, DE ) with a shear rate range from 0.1 to 1000 s -1 at measuring temperature of 20 °C.

## GEL STRENGHT

- Adapted standard BLOOM method with a TA XT2 Texture Analyzier (Stable Mirco Systems, Godalming, UK)
- A plunger (Ø 0,5 in) was pressed 4 mm deep in the surface of the sample (without breaking)

## GELATION SPEED AND MELTING POINT

- The measurements were performed with a plate-plate (Ø 4cm) oscillation rheometer (AR 2000, TA Instruments, New Castle, DE )
- Parameter chosen: plate gap - 1000 µm, torque - 0,001 Nm, frequency - 1 Hz.

# RESULTS & DISCUSSION

1. APPARENT VISCOSITY
2. IMPACT OF UHPH AT pH 3 AND pH 6 ON PECTIN MOLECULAR CHARACTERISTICS
3. GEL STRENGTH
4. GELATION SPEED AND MELTING POINT

# 1. APPARENT VISCOSITY

- AU and CU pectin were UHPH treated at 50, 150 and 250MPa
- CU pectin had higher apparent viscosity than AU pectin
- UHPH caused more pronounced decrease in apparent viscosity of CU pectin then in AU pectin (inline with previous studies)

(a)

(b)

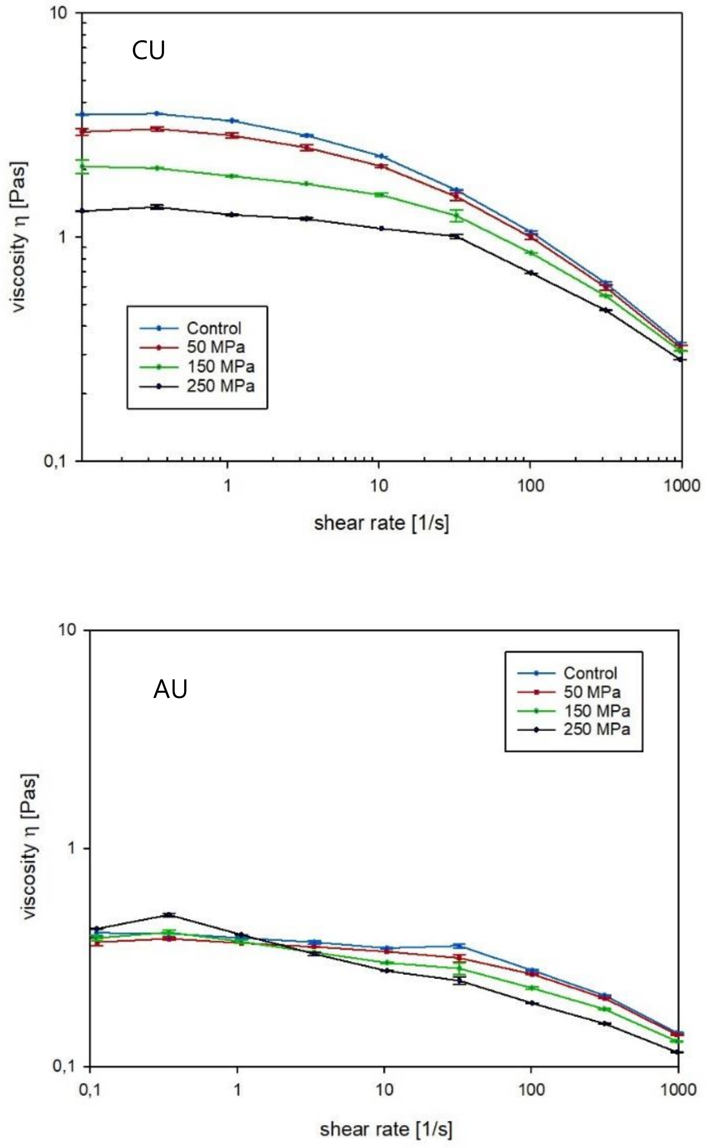



*Figure 1. Apparent viscosity of untreated and UHPH treated (a) CU and (b) AU pectin solution.*

# 1. APPARENT VISCOSITY

- Both, untreated and UHPH treated AU & CU pectin exhibited shear thinning behavior (inline with previous studies of Chen et al. (2012) & Xie et al. (2018))
- With increasing pressure: ↓ consistency index (K) & ↑ flow index (n) observed for CU & AU pectin.
- Consistency index of untreated and UHPH treated CU samples significantly differed (Tukey test, p<0,001).

*Table 1. Flowing properties of CU and AU pectin solution*

| Sample | Consistency index K [Pas$^n$] | Flow index n [-] | $R^2$ [-] |
|---|---|---|---|
| CU untreated | 8,659 ± 0,236 | 0,534 ± 0,004 | 0,996 |
| CU 50 | 7,914 ± 0,23 | 0,542 ± 0,005 | 0,995 |
| CU 150 | 5,507 ± 0,154 | 0,590 ± 0,005 | 0,996 |
| CU 250 | 3,710 ± 0,098 | 0,633 ± 0,004 | 0,997 |
| AU untreated | 0,337 ± 0,005 | 0,816 ± 0,002 | 0,999 |
| AU 50 | 0,291 ± 0,004 | 0,829 ± 0,002 | 0,999 |
| AU 150 | 0,218 ± 0,004 | 0,855 ± 0,003 | 0,999 |
| AU 250 | 0,171 ± 0,003 | 0,871 ± 0,003 | 0,999 |

(a)

(b)

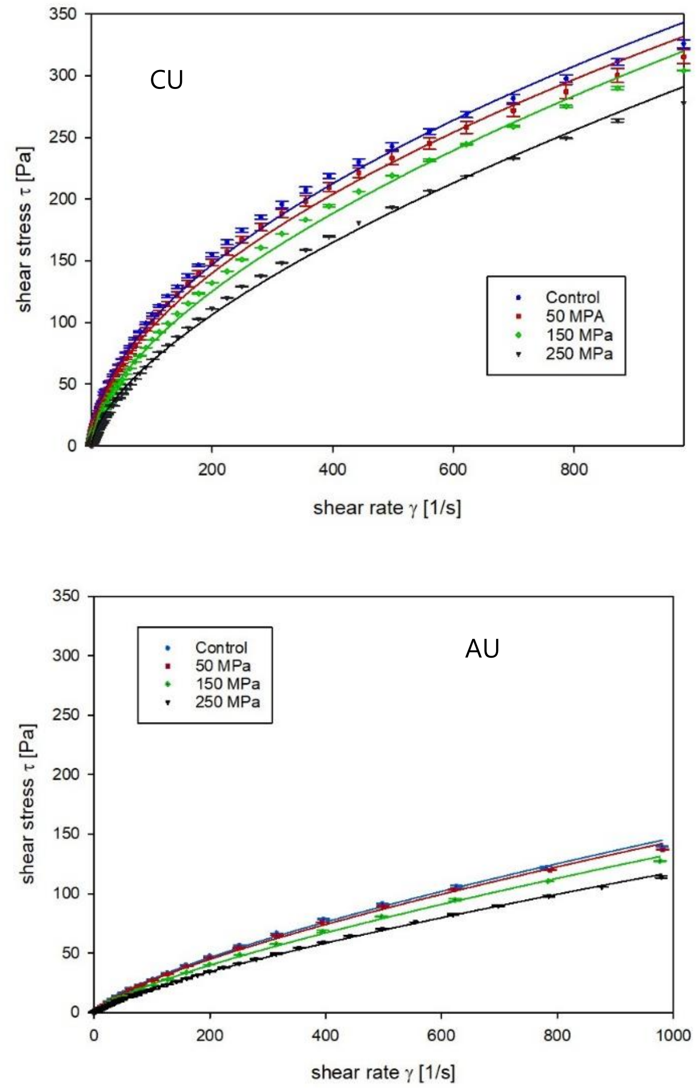



*Figure 2. Flowing properties of untreated and UHPH treated (a) CU pectin and (b) AU pectin.*

# 2. IMPACT OF UHPH AT pH 3 & pH 6 ON PECTIN MOLECULAR CHARACTERISTICS

- AU and CU pectin were treated at 100, 200 and 300 MPa
- Average MW of the pectins differed depending on the source and the pH value (Figure 3A and B).
- CU showed the pressure dependent decrease, that was much less pronounce in AU.
- The dispersity (i.e. Mw/Mn, Figure 3 E, F) was much more impacted by UHPH pressure in AU pectin, suggesting that the degradation of only larger chains of pectin had a larger impact for this pectin.

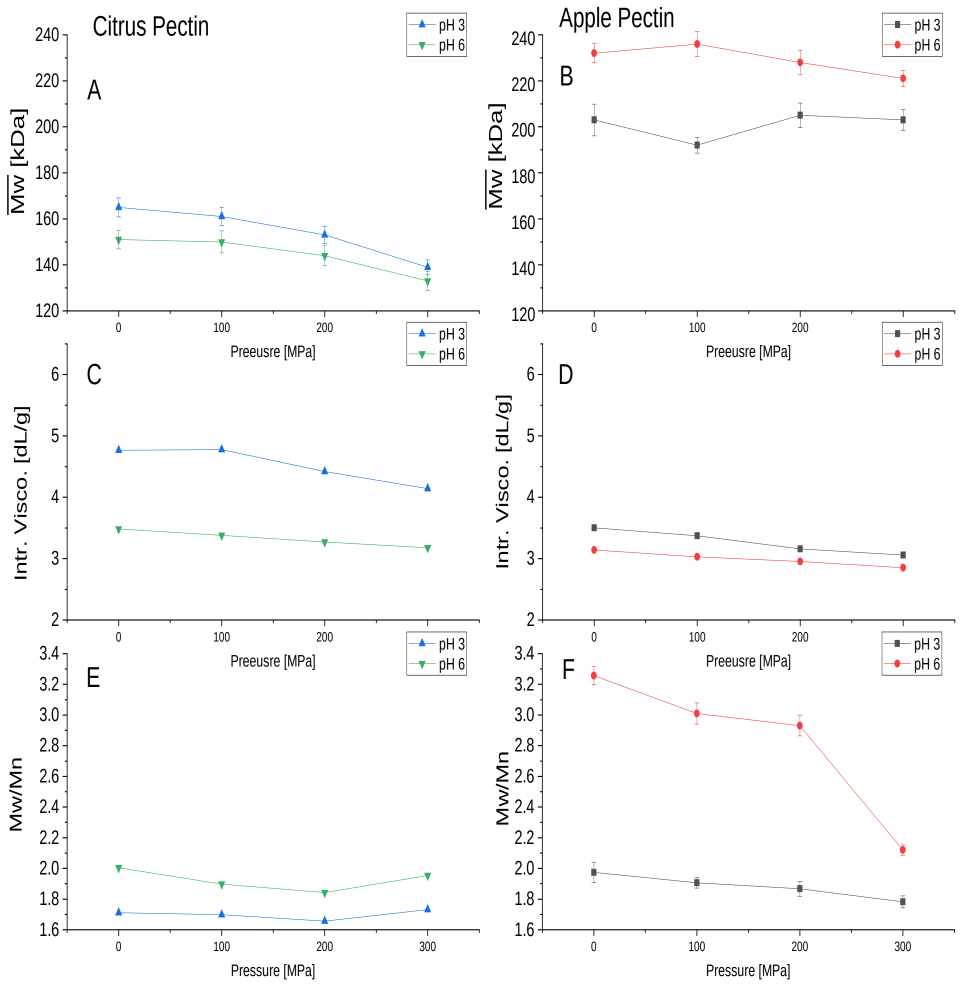
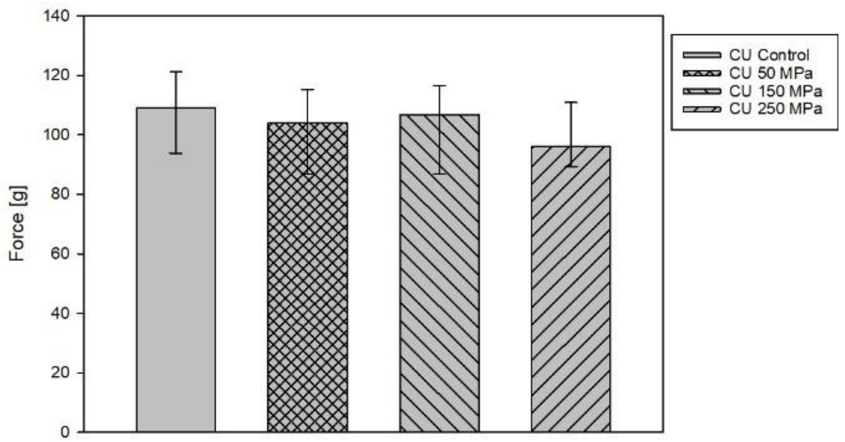



*Figure 3. The impact of HPH at 3 levels (0.1 MPa, 100 MPa, 200 MPa, 300 MPa) on the mass-weighted average molecular weight (A, B), intrinsic viscosity (C, D) and dispersity (E, F) of citrus (CU) pectin (A, C, E) and apple (AU) pectin (B, D, F) when treated at pH 3 and pH 6.*

# 3. GEL STRENGHT

- CU pectin forms stronger gels than AU pectin, likely due to a larger content of neutral sugars in apple pectin compared to citrus one (Kravtchenko et al. (1992)).
- Although statistically significant differences were found between apparent viscosity of untreated and UHPH treated pectin samples, those differences were not observed in strength of the produced pectin gels.
- UHPH treatment did not have a statistically significant impact on gel strength of AU ($p = 0,462$) and CU ($p = 0,771$) under examined conditions.

(a)




(b)

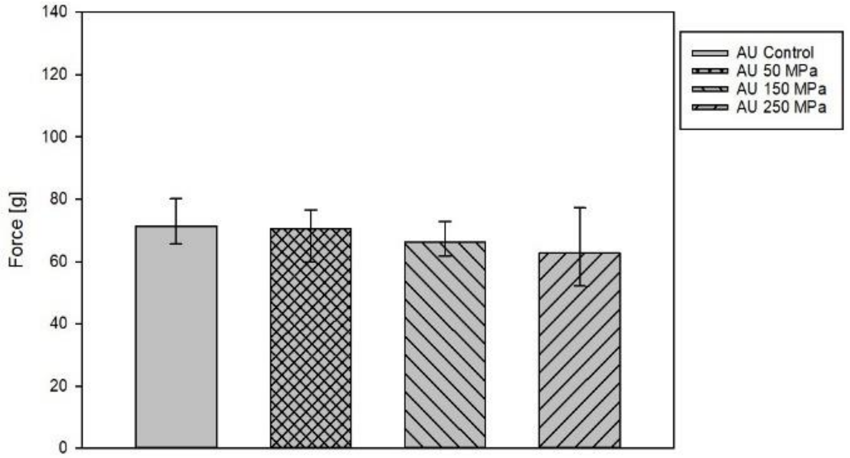


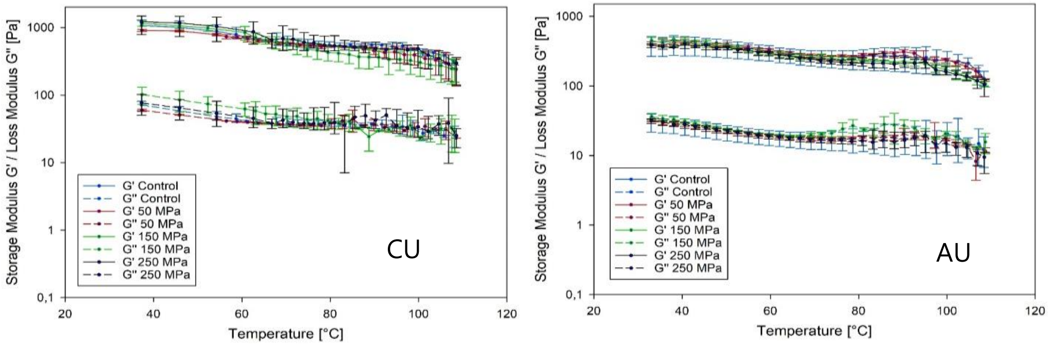

*Figure 4. Gel strength of untreated and UHPH treated (a) CU and (b) AU pectin.*

# 3. GELATION SPEED & MELTING POINT

- All pectin gel samples exerted non-melting behavior (Figure 5).
- UHPH CU pectin samples had shorter gelation time (higher gelation temperatures) (Table 2; Figure 6)
- Statistically significantly differences not found.

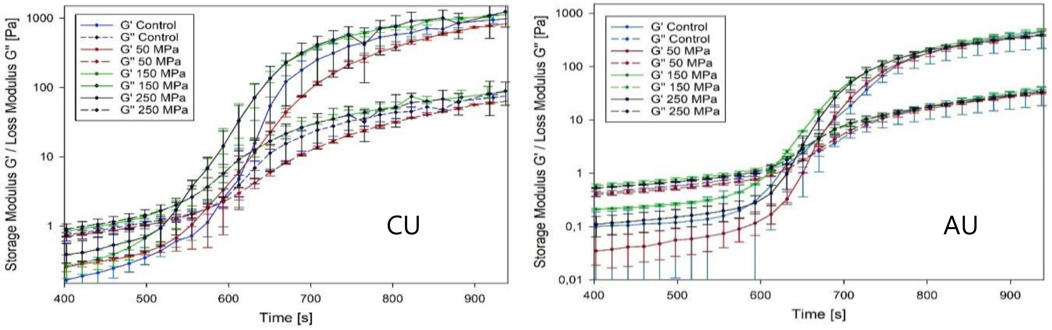
*Table 2. Gelation temperature and speed of untreated and HPH treated CU & AU pectin*

| Indicators | Sample name | | | |
|---|---|---|---|---|
| | **CU Untreated** | **CU 50** | **CU 150** | **CU 250** |
| Gelation Temperature [°C] | 59,2 ± 1,1 | 63,2 ± 1,2 | 62,4 ± 0,9 | 62,5 ± 1,0 |
| Gelation time [s] | 586,4 ± 14,6 | 572,1 ± 28,1 | 547,7 ± 10,5 | 546,9 ± 13,3 |
| | **AU Untreated** | **AU 50** | **AU 150** | **AU 250** |
| Gelation Temperature [°C] | 54,8 ± 1,7 | 52,1 ± 0,1 | 57 ± 0,4 | 54,7 ± 0,5 |
| Gelation time [s] | 639,2 ± 21,0 | 656,5 ± 2,6* | 616,4 ± 2,1 | 644,6 ± 2,8 |




(a) (b)

*Figure 5. Storage Modulus G' and Loss Modulus G'' of untreated and UHPH treated pectin (a) CU and (b) AU gels during heating step.*




(a) (b)

*Figure 6. Storage Modulus G' and Loss Modulus G'' of untreated and UHPH treated pectin (a) CU and (b) AU samples during cooling and gelation step.*

# CONCLUSION

From the obtained results, it can be concluded that UHPH process has a potential to modify the structure and flowing behavior of pectins, but further research is needed in order to elucidate all the changes and potential benefits of this ubiquitous and multifunctional hydrocolloid treated by UHPH.

# THANK YOU VERY MUCH FOR YOUR ATTENTION

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