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Towards in-line rheology measurement of protein melts during high moisture extrusion by pulsed ultrasound velocimetry

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ABSTRACT

The present paper is concerned with the development of a custom pulsed ultrasound velocity profiling (PUV) methodology to non-invasively measure, analyze and control protein melt flow and power-law flow indices in the cooling die during high moisture (HME) extrusion processing. The methodology is first validated on glycerol and a carboxymethyl cellulose (CMC) solution as Newtonian and shear-thinning reference fluids, respectively, at different flow rates followed by application to two pea protein melts at different moisture contents (MCs) in the low flow rate regime typical of a pilot-scale extruder and characterized by a poor signal to noise ratio (SNR) close to the extruder die wall. The flow indices were compared with those obtained from conventional rheometry, showing good agreement for the reference fluids and semi-quantitative agreement for the protein melts. The study confirms that PUV can be used for in-line application in a cooling die by measuring the local flow conditions, as well as contribute to the understanding of protein melt fibre formation. On the other hand, the SNR close to the die wall need to be improved e.g. by using an ultrasound transducer operating at higher frequency and modifying the die to enable quantitative agreement with flow simulations to properly extract local rheometric data. Thus, it is concluded that further refinement of the methodology is both possible and needed to improve the accuracy of the measurements in future work for in-line application during HME extrusion.

1. Introduction

Rheological properties of viscoelastic materials such as food, cosmetics, and polymers are used to monitor the quality, process, and performance of unit operations. High moisture extrusion (HME) is one of the main unit operations used to produce texturized and fibrous structured meat analogues (HMMA) from plant proteins. Exposures to high thermomechanical stresses combined with the physico-chemical conditions in the cooling die, deforms, fractionates, and reorganizes the original plant protein structures, resulting in anisotropic, meat-like structures. While these meat-like structures are created by optimizing processing parameters and regulating the formulations, the mechanisms behind fibre formation during HME are still not fully elucidated. However, many researchers have contributed to revealing the mechanisms of fibre formation in protein melts (Kaunisto et al., 2024; Sandoval Murillo et al., 2019; van der Sman and van der Goot, 2023; Wittek et al., 2021a, 2021b, 2021c; Zink et al., 2024). The rheological properties of such materials are generally measured through offline instruments and the results are then used to interpret material behavior under processing conditions. On the other hand, such measurements cannot be performed under the combination of high temperature and high pressure that exists in an extruder. Inline measurements of the rheological properties therefore provide more accurate and precise data control over the process.

Pulsed ultrasound velocity profiling (PUV) systems coupled with pressure difference (PD) measurements are used for real time measurements of rheological properties (Kotzé et al., 2012, 2013; Takeda, 1986, 1995; Wiklund et al., 2007) and are available as commercial systems, for example by Incipientus, as used in the present study. These systems provide spatial, real time, instantaneous velocity profiles across the measurement axis, originally based on the Doppler frequency shift observed due to the moving fluid particles (Kotzé et al., 2011; Takeda, 1986, 1991; Wiklund et al., 2007). As the rheological properties of fluids may depend on the local flow conditions, real time measurements of

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instantaneous velocity profiles across the fluid conduit are key to properly understand the flow behavior in such systems. Accurate rheological measurements can provide insights into the viscous and elastic behavior of fluids, which are essential for predicting how materials will flow through the extruder and how fibres can be formed. Understanding these properties allows to fine tune process parameters, such as temperature, pressure, and screw speed, ensuring a stable and consistent extrusion process. Measuring rheological properties under extrusion conditions is difficult with offline measurement systems, mainly due to the challenges with replicating the extrusion conditions (geometry, shear, dynamics etc.) and loss of moisture above 100 °C. Therefore, measuring the rheological properties under extrusion-relevant conditions is crucial for gaining insights into the real time flow behavior.

The application and technology of PUV has been developing significantly over the last years. Takeda Y, 1986, pioneered the use of the ultrasonic Doppler frequency shift method to measure the velocity profile of a fluid in one dimension (Takeda, 1986). Many researchers developed tools and methods to measure rheological properties of fluids by combining PUV with PD measurements for consistent flows along the measurement axis (Han et al., 2002; Morgen et al., 2005; Ouriev and Windhab, 2007; Wiklund et al., 2007). The PUV system has the advantages of being non-invasive, easy to install, portable, and works for all kinds of fluids, including opaque fluids, compared with other velocity profiling methods (Kotzé et al., 2011; Powell, 2008; Teufel et al., 1992). The PUV technique has been tested as an effective tool for real time measurements of rheological properties of viscoelastic materials, such as cellulose pulp, yoghurt, marmalade, fruit soup (Wiklund and Stading, 2008), chocolate (Birkhofer et al., 2008), fat blends (Wassell et al., 2010), tomato ketchup (Berta et al., 2016), polydimethylsiloxane (PDMS) (Dogan et al., 2005), mayonnaise, xanthan solution and pasta sauce (Wiklund et al., 2007). The rheological properties are evaluated by fitting the measured velocity profiles with the appropriate rheological model, where the additional pressure drop data can be used to evaluate the shear stress.

To our knowledge, there are few publications in the literature applying PUV to extrusion processing for monitoring the flow velocity profiles (Putz et al., 2014) and none measuring non-invasively. Since the flow behavior during extrusion is inherently hidden from view with high temperature and pressure, applying PUV has a great advantage over other techniques to monitor and measure rheological parameters in-line. Thus, the PUV measurement process provides a much-needed understanding to monitor the overall process and mechanisms during extrusion. Moreover, comparing the experimental PUV measurements with conventional rheometry shows good agreement in the high flow rate regimes with good signal-to-noise ratios (Wiklund et al., 2007; Young et al., 2008).

The low flow rate regime, in this paper characterized by a poor signal to noise ratio (SNR) close the extruder die wall, present significant challenges in accurately measuring velocity profiles by PUV, making it difficult to obtain precise measurements (Roshanaei et al., 2022; Shen and Guo, 2022). Moreover, low flow velocities can result in reduced Doppler frequency shifts, making it difficult to distinguish between fluid movement and background noise, which further complicates the detection of wall positions.

The aim of this study was to develop a method for the real time low flow-rate measurement of extruded protein melts by PUV and to validate them with results obtained by conventional rheometry. Experiments were performed using reference fluids with known rheological behavior (Newtonian and shear thinning) and on protein melts in high moisture extrusion. The rheological properties of the corresponding materials were determined using conventional rheometry and the results were compared with those obtained from PUV, using a custom methodology complementary to the ones provided in the Incipientus software.

2. Materials and methods

2.1. Materials

Pea protein isolate (86% protein, Pisane™ M9) and pea fiber (Swelite™) was purchased from Cosucra (Lestrem, France and Warcoing, Belgium). Food grade glycerol (99% purity) was obtained from Rawfoodshop Scandinavia AB as a Newtonian reference fluid, and carboxymethyl cellulose (CMC) was acquired from Engelhardt (Göteborg, Sweden) to form a 2% CMC shear-thinning reference fluid. The protein samples were stored in moisture and temperature-controlled rooms until further use and analysis.

2.2. Extrusion experiments and sample preparations

A twin-screw extruder with screw diameter 20 mm and length to diameter ratio, L/D, of 40:1 was used (Brabender TwinLab-F 20/40 D, Duisburg, Germany). The extruder had four heating zones and a separate die heating element. The extrusion parameters were selected based on previous trails and studies. The screw speed and feeding rate were set to 400 rpm and 4 kg/h, respectively, while the barrel temperatures were set to 40 °C in the hopper zone, 60 °C in the mixing zone, 120 and 130 °C in the melting zones and 130 °C in the die section. In the die section, temperature and pressure were measured by using the corresponding sensors. At the end of extruder die, a long rectangular cooling die (600 mm × 30 mm × 5 mm) was connected, with its temperature set at 60 °C.

To prepare samples for offline rheological measurements of the protein melts, the die and melting zones temperatures were set to 100 °C. Pea protein isolate and pea fibers in an 85:15 ratio were thoroughly mixed immediately before extrusion and the samples were then extruded into a 13 mm cylindrical die without connecting the cooling sections. In the extruder, water was added to obtain a MC of either 60 or 64% by adjusting the solid feeding rate (kg/h) and water pumping rate (rpm). The moisture content of the extrudate and mixed powder were analyzed gravimetrically by drying at 105 °C over night. The procedure ensured well mixed samples to simulate the extrusion conditions of

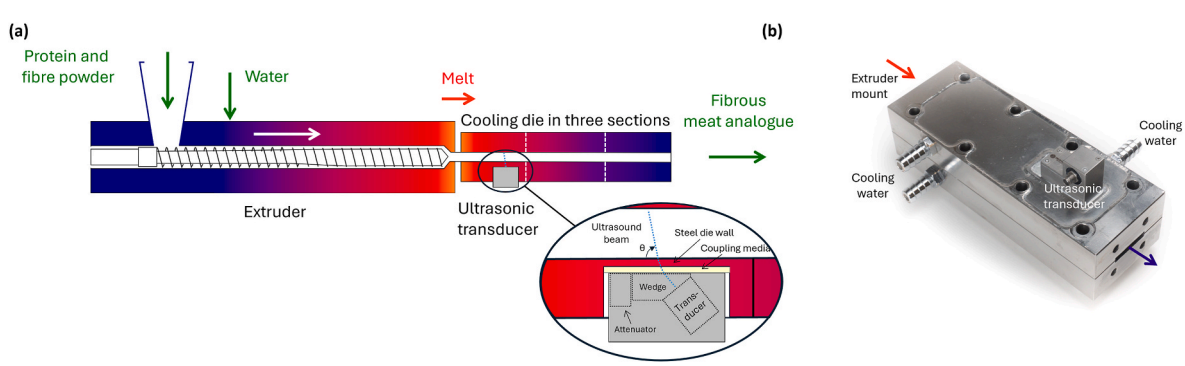

Fig. 1. (a) Schematic illustration of the extruder and transducer setup. (b) Photo of the cooling die with the mounted PUV transducer and indicated flow direction.

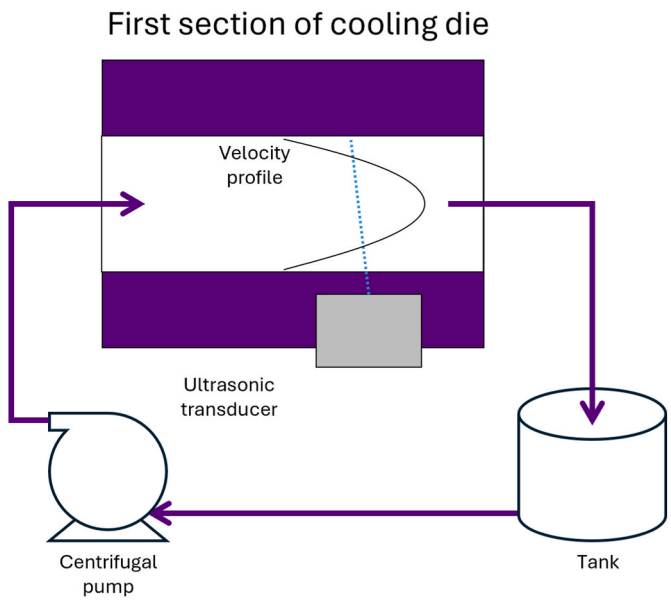

Fig. 2. Experimental setup for cellulose and glycerol velocity profile measurement.

protein melt in the die during measurements. The original MCs of 60% and 64% in the extrudates were reduced to 49% and 55%, respectively, during extrusion due to evaporation. The extruded samples were stored cold at 4 °C in plastic bags to mitigate MC loss followed by rheological and MC analysis of the mix the next day.

2.3. PUV experimental setup and techniques

The PUV measurements were done in a 200 mm long stainless-steel section of the cooling die with a rectangular flow channel cross-section of 5 mm height and 30 mm width. The ultrasound transducer was mounted at a 150 mm distance from the fluid cooling die inlet in a custom designed recessed well of rectangular geometry. The transducer was installed in the bottom section and measured the fluid velocity profiles along the height dimension of the channel. The rectangular die was then connected to either an extruder for measuring pea protein melts or a pump for measuring the velocity profiles of the 2% CMC solution and glycerol as references. During extrusion of the protein melts, the temperature of the cooling die was maintained at 60 °C by circulating water. For CMC and glycerol, the flow in the cooling die was instead driven by a low-pulsation centrifugal pump connected to a tank containing 10 L of the fluids at temperatures of 53 and 55 °C, respectively, with the temperature monitored every 5 min by a thermocouple. Schematic illustrations of the PUV experimental setups for extrusion and pumping are shown in Fig. 1 and Fig. 2.

2.4. PUV principle and measurements

The Incipientus in-line flow visualizer (IFV) from Incipientus Ultrasound Flow Technologies AB, Gothenburg, Sweden, with a custom designed non-invasive Incipientus transducer and software was used to record the instantaneous velocity profile along the pulsed beamline. A series of consecutive pulsed ultrasonic waves at fixed frequency repetitions were emitted into the cooling die through the stainless-steel wall, see Fig. 1. The instrument then switched to receiver mode to collect the returning echo during the time between successive pulses. The principle of PUV is explained in detail elsewhere (Evans and McDicken, 2000; Takeda, 1986; Wiklund et al., 2007). In this study, the ultrasound base frequency was 4 Mhz, with 6 cycles per pulse and a Doppler angle of 68°. The sound velocities in the respective fluids were taken from literature as 1900 m/s, 1540 m/s and 1540 m/s for glycerol, CMC and pea protein (assumed similar to muscle protein), respectively (Ahmadi et al., 2023; Gyöngy and Kollár, 2015; Punitha et al., 2014; Shin et al., 2010). One single velocity profile took about 300 ms to record and 127 single velocity profiles were collected and averaged automatically in the Incipientus software. In this way, 30 different velocity profile data sets were obtained for further processing.

The PUV instrument used a fast Fourier transform (FFT) algorithm to analyze the received echo and then to evaluate the instantaneous velocity of the fluid at each point from the Doppler frequency shift. The distance between each measurement point at a particular depth is evaluated by the time of flight of the ultrasound waves and the reflected echo, i.e. in both directions where the measurement points are indexed as consecutive gates. The distance between gates is given by equation (1)

$$\Delta r = \frac{D_f c \sin(\Theta)}{2F_{SC}} \tag{1}$$

where Δr is the distance between gates, D_f is the decimation factor, c is the sound velocity in m/s, Θ is the Doppler angle in degrees and F_{SC} is the system clock frequency of the field programmable gate array (FPGA) board in Hz.

By measuring the Doppler shift in the frequency of the returned signal, the velocity of the fluid at different points along the path can be determined by equation (2)

$$v = \frac{c\Delta f}{2f\cos(\Theta)} \tag{2}$$

where v is the velocity of the fluid at each gate, c is the ultrasound speed in m/s, Δf is the Doppler frequency shift in Hz, f is the emitted ultrasound frequency in Hz.

2.5. PUV post-data collection processing

The low flow-rate regime studied in the present paper poses challenges on data collection and processing to obtain reliable velocity profiles, due to the relatively unstable flow inside the extruder cooling die. A custom methodology was therefore applied where the echo signals collected from the individual experiments by the Incipientus instrument and software (Wiklund et al., 2007) were further processed by first stacking the 127 individual velocities for all gates in the 30 experimental data sets for a given fluid/melt into a 2D matrix in MATLAB (The MathWorksInc., 2022). Each column of the 2D matrix, corresponding to all velocity data at a particular gate, was then min-max sorted and the five percentile was symmetrically removed by default to minimize the effect of outliers. The remaining data points were then used to estimate the mean and standard deviations for the flow velocities at all gate positions.

2.6. Rheological measurements

Small amplitude oscillatory shear (SAOS) tests were measured on a HR-30 rheometer equipped with temperature controlled 25 mm parallel plates (TA instrument, New Castle, USA). Samples with a thickness of 2 mm were prepared from the 13 mm diameter protein melt extrudates using a vacuum holder (Stading and Langer, 1999). The samples were then placed in the measuring gap and a compression force of 5 N was applied while heating the samples from 25 to 40 °C. The axial compression force was then reduced to 1 N at 40 °C and kept throughout the measurements. Mechanical spectra were recorded from 0.1 to 30 Hz at constant temperatures of 40, 50, 60, 70, 80 and 90 °C with heating rate 10 °C/min between the temperature steps. Paraffin oil was applied to the edges of all samples during measurements to reduce the impact of evaporation at elevated temperatures. Cox-Merz rule was assumed valid and the complex viscosity at a specific angular frequency was used corresponding to a shear viscosity at a shear rate equal to the angular frequency.

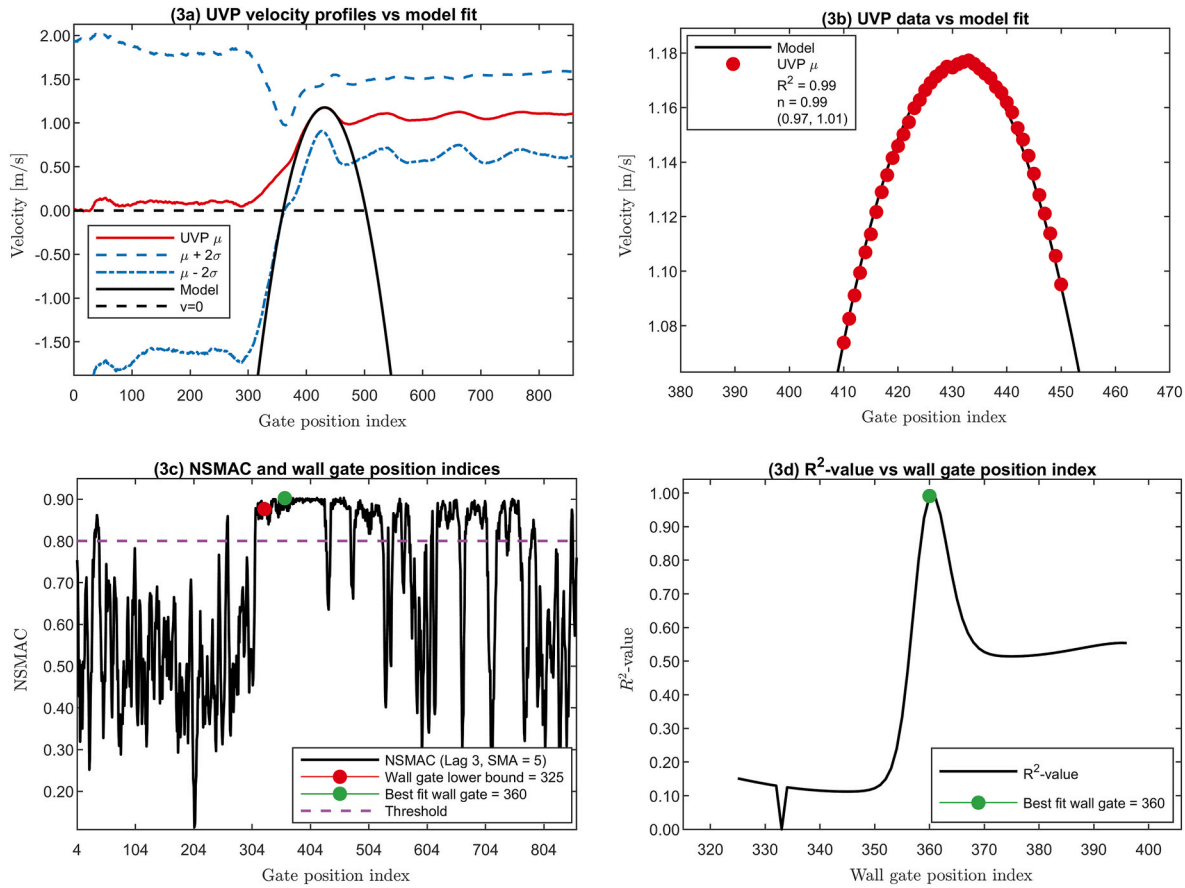

Fig. 3. (a) PUV velocity data with mean (μ), standard deviation (σ), the fitted model and zero velocity line for glycerol. (b) PUV mean data, fitted model near the velocity maximum, R^2-value, flow behavior index and associated 95% confidence interval. (c) NSMAC curve with the wall gate lower bound index, threshold value and best fit wall gate index. (d) R^2-value and best fit wall gate index.

The shear viscosity of glycerol and CMC was characterized at the temperatures used for the PUV measurements, at shear rates between 0.01 and 100 s^{-1}, using an ARES-G2 rheometer (TA Instruments, New Castle, USA) with concentric cylinders geometry (27.7 mm bob diameter and 30 mm cup diameter). The cup rotated and the bob remained stationary. About 22 mL of sample was loaded and left for temperature stabilization before starting the measurements. All rheological measurements were performed in triplicates.

3. Theory and calculation

3.1. Velocity profile assumptions

Due to the 6:1 width to height aspect ratio of the extruder die cross-section, the flow was modeled using the parallel plate assumption analogous to the previously investigated cylindrical situation (Wiklund et al., 2007), effectively reducing the Navier-Stokes equations to equation (3) with the assumed power-law constitutive model in equation (4),

$$\frac{d\tau}{dy} = \frac{dp}{dx} \tag{3}$$

$$\tau = K\left(\frac{dv_x}{dy}\right)^n \tag{4}$$

where τ is the shear stress, p is the fluid pressure, y is the coordinate in the height direction from the die center, x is the coordinate in the length direction, v_x is the velocity in the length direction, K is the consistency index and n is the flow behavior index. Combining equations (3) and (4) with standard no-slip and symmetry assumptions at the die wall and center, respectively, yields the velocity profile, equation (5),

$$v_x(y) = d^{\frac{n+1}{n}}\left(\frac{1}{K^{1/n}}\right)\left(\frac{n}{n+1}\right)\left(\frac{dp}{dx}\right)^{\frac{1}{n}}\left(1 - y^{*\frac{n+1}{n}}\right) = v_{max}\left(1 - y^{*\frac{n+1}{n}}\right) \tag{5}$$

where d is the half height of the channel, y^* is the dimensionless distance and v_{max} is the maximum velocity. y^* can also be written in terms of the relative gate position index ratio by assuming a wall gate position index and calculating the corresponding flow velocity vertex gate position index based on the half height of the channel and a distance-to-gate scaling parameter obtained from the PUV system software, as given in equations (6) and (7),

$$y^* = \frac{GPI_{vertex} - GPI}{GPI_{vertex} - GPI_{wall}} \tag{6}$$

$$GPI_{vertex} = GPI_{wall} + \left(\frac{\Delta GPI}{\Delta y}\right)d \tag{7}$$

where GPI is the gate position index, GPI_{vertex} is the flow velocity vertex gate position index, GPI_{wall} is the wall gate position index and $\frac{\Delta GPI}{\Delta y}$ is the gate-to-distance (GTD) scaling parameter.

3.2. Wall position estimation

Due to the low ultrasound SNR close to the extruder die wall combined with the joint effects of ultrasound pulse size vs geometry size and overlapping pulses near the wall, obtaining a reliable estimate of the wall gate position index is not straightforward. Thus, to be able to obtain a lower bound estimate for the model fitting procedure, the moving

Table 1
Comparison between flow behavior indices obtained from conventional rheometry with mean, minimum and maximum values and PUV data fitting with mean and 95% confidence interval.

Sample	Temperature (°C)	* ***n***-value from rheometry data	** ***n***-value from PUV data
Glycerol	55	1.0 (1.0, 1.0)	0.99 (0.97, 1.0)
CMC 2%	53	0.51 (0.50, 0.51)	0.53 (0.52, 0.54)
Pea protein at *49% and **60% MC	60	0.09 (0.09, 0.09)	0.22 (0.21, 0.22)
Pea protein at *55% and **64% MC	60	0.09 (0.09, 0.09)	0.17 (0.16, 0.17)

autocorrelation (MAC) with a window size of every four consecutive gates and a lag of three gates in each window, respectively, was calculated from the average velocity profile for each fluid. The MAC curve was then smoothed with a centered moving average (SMAC) defined on five consecutive gates to filter out the characteristics of the wall-fluid transition region. To enable simple thresholding, the absolute value of the SMAC curve was calculated and further normalized with its maximum value to yield a feature curve (NSMAC). The NSMAC curve was finally used to find the first point where the mean value of the NSMAC curve for the current gate and the 19 previous gates rise above a threshold value of 0.8, thereby yielding the lower bound estimate. The mentioned window size, lag, smoothing, averaging and thresholding parameter values were all obtained by simple trial and error to maintain a balance between sensitivity, smoothness and the ability to capture rapid correlation changes in the resulting NSMAC curve.

3.3. Parameter fitting procedure

The NSMAC curve was calculated to obtain the first lower bound wall gate position index and the model, given by equations (5)–(7), was then used to fit the flow behavior index and maximum velocity parameters to PUV data chosen with an upper and a lower bound close to the maximum velocity for each fluid. The coefficient of determination (R^2-value) for the current wall position was then stored and the procedure was repeated by stepping through all subsequent gates up to a wall gate corresponding to the vertex point associated with the initial wall position. The fitted parameters corresponding to the maximum R^2-value were then chosen as the optimal fit and the resulting flow behavior index was validated against the corresponding value obtained from conventional rheometry.

4. Results

4.1. Glycerol

PUV data from glycerol as a Newtonian reference fluid at a maximum velocity around 1 m/s was studied. As can be seen in Fig. 3a, the velocity profile demonstrates low correlated noisy data in the extruder die wall with high standard deviations up to the wall-fluid transition region, where the standard deviation drops to give a distinct increase in SNR. Fig. 3b further shows that the PUV mean data around the maximum experimental velocity are enough to obtain a flow behavior index estimate consistent with the corresponding rheometry measurement, see Table 1. Care was taken to not include PUV data in the low SNR region close to the wall, as well as to deep in the fluid where the signal scattering effects result in a flat velocity profile. The conservative initial

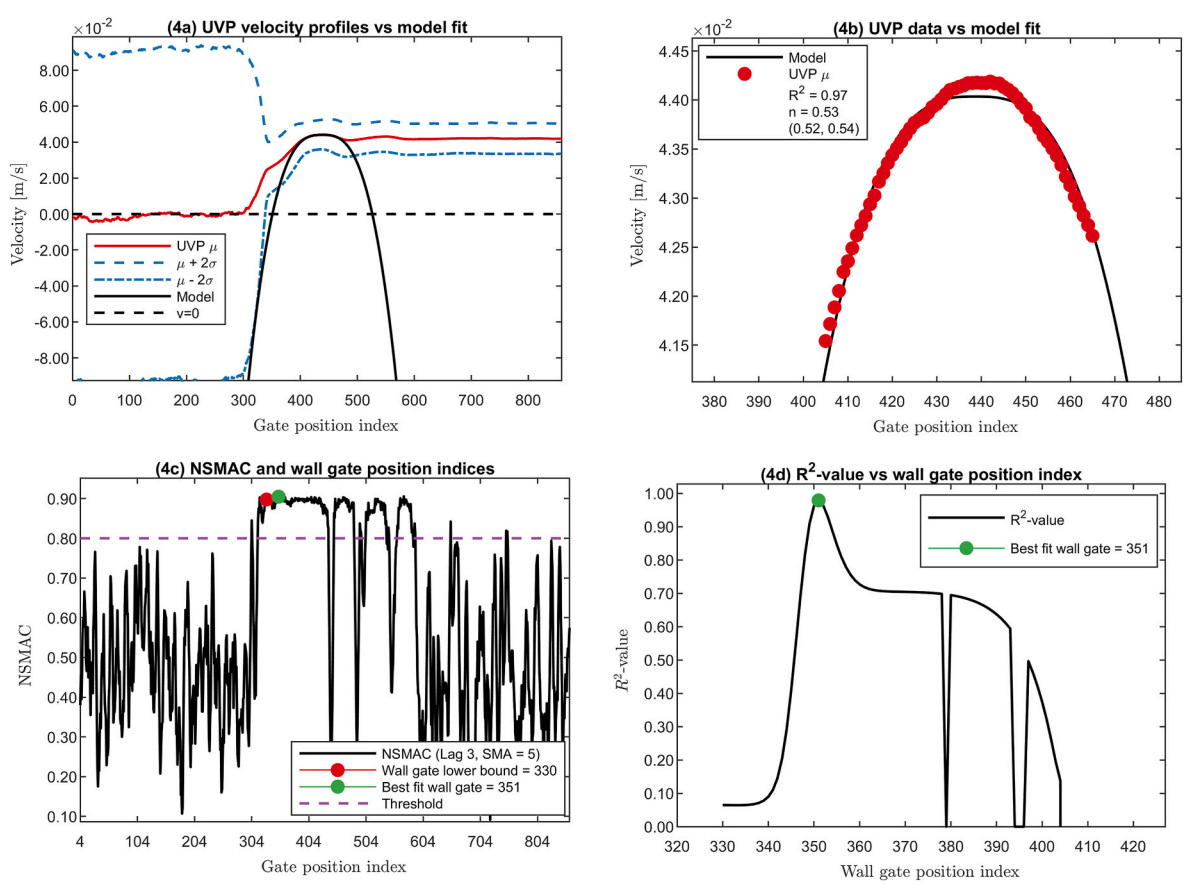

Fig. 4. (a) PUV velocity data with mean (μ), standard deviation (σ), the fitted model and zero velocity line for CMC. (b) PUV mean data, fitted model near the velocity maximum, R^2-value, flow behavior index and associated 95% confidence interval. (c) NSMAC curve with the wall gate lower bound index, threshold value and best fit wall gate index. (d) R^2-value and best fit wall gate index.

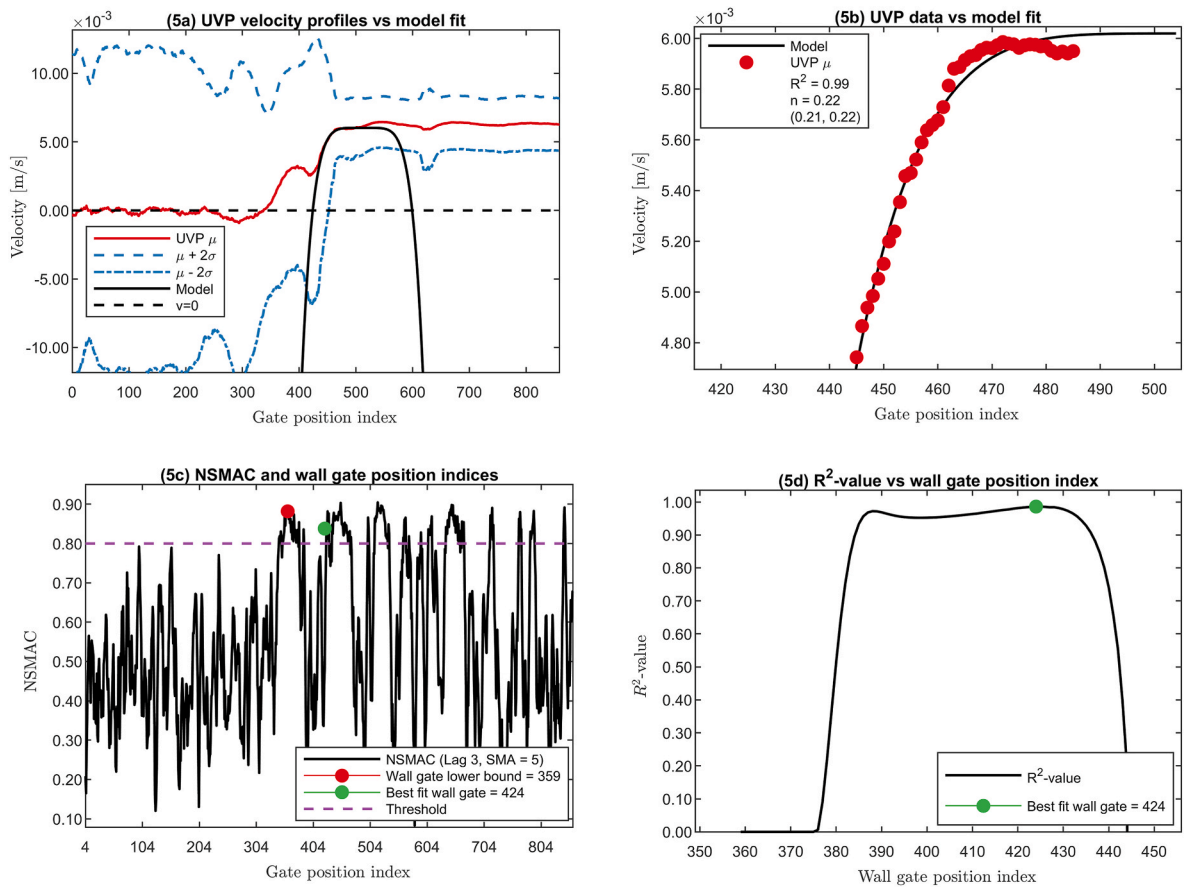

Fig. 5. (a) PUV velocity data with mean (μ), standard deviation (σ), the fitted model and zero velocity line for protein melt at 60% MC. (b) PUV mean data, fitted model near the velocity maximum, R^2-value, flow behavior index and associated 95% confidence interval. (c) NSMAC curve with the wall gate lower bound index, threshold value and best fit wall gate index. (d) R^2-value and best fit wall gate index.

estimate of the lower bound wall gate position index of 325 is also confirmed in Fig. 3c by the behavior of the NSMAC curve to the right and left of this point, showing consistently and inconsistently correlated distinct regions, respectively. The best fit wall gate position index of 360 is also at a point where the small remaining noise in the NSMAC curve is almost canceled out and supported by a well-defined R^2-value peak, as shown in Fig. 3c-d.

4.2. CMC

In the case of shear-thinning CMC, the SNR showed a similar pattern to glycerol with a high standard deviation in the wall that distinctly drops in the wall-fluid transition region, as can be seen in Fig. 4a. On the other hand, the fit to the PUV data in Fig. 4b shows somewhat less good agreement with experimental data, although resulting in a flow behavior index close to that obtained from rheometry, see Table 1. Fig. 4c further confirms that the NSMAC curve shows a similar pattern to glycerol with a lower bound wall gate index at 330 and a best fit wall gate index at 351 supported by a well-defined R^2-value peak in Fig. 4d. However, a difference compared with glycerol is that the wall position does not seem to coincide with the "bump" minimum of the $\mu + 2\sigma$ curve, instead being at a slightly higher gate index, which illustrates the ambiguity in determining the wall position from simple velocity profile characteristics. As CMC shows a flatter velocity profile compared to glycerol, a careful selection of lower and upper bounds for the experimental PUV data used in the model fit needs to be made to avoid the potential adverse influence of low SNR and signal scattering effects.

4.3. Protein melt

The results from two protein melts at 60 and 64% MC showed a similar behavior and decent agreement with the PUV data in Fig. 5a-b and Fig. 6a-b , respectively, although the PUV mean data show more pronounced irregularities and lower SNRs in the velocity profiles compared to glycerol and CMC. The low SNRs are also seen in terms of the non-consistent correlations shown in the NSMAC curves in Fig. 5c and Fig. 6c , where the wall gate transitions are less distinct, giving lower bound wall gate indices of 359 and 365, respectively. Given the similarity of the samples, the estimates for the lower bound wall gate indices seem to be rather consistent and work reasonably well, but due to the maintained inconsistent correlations beyond this point, it is harder to find a distinct best fit wall gate. The latter is also confirmed by the relatively flat R^2-value landscapes in Fig. 5d and Fig. 6d, yielding best fit wall gate indices of 424 and 429, respectively, although with many fits showing relatively high R^2-values. The resulting PUV data estimates of the flow behavior indices show larger but reasonable discrepancies with those obtained from conventional rheometry in Table 1. Since all phenomena were likely not accounted for and given the current variations in MCs, the agreement is deemed semi-quantitative.

5. Discussion

5.1. Agreement between in-line PUV and conventional rheometry

For both glycerol and CMC, the validity of the present approach can be confirmed by the flow behavior indices from conventional rheometry and in-line PUV being in reasonable agreement. For unstable protein

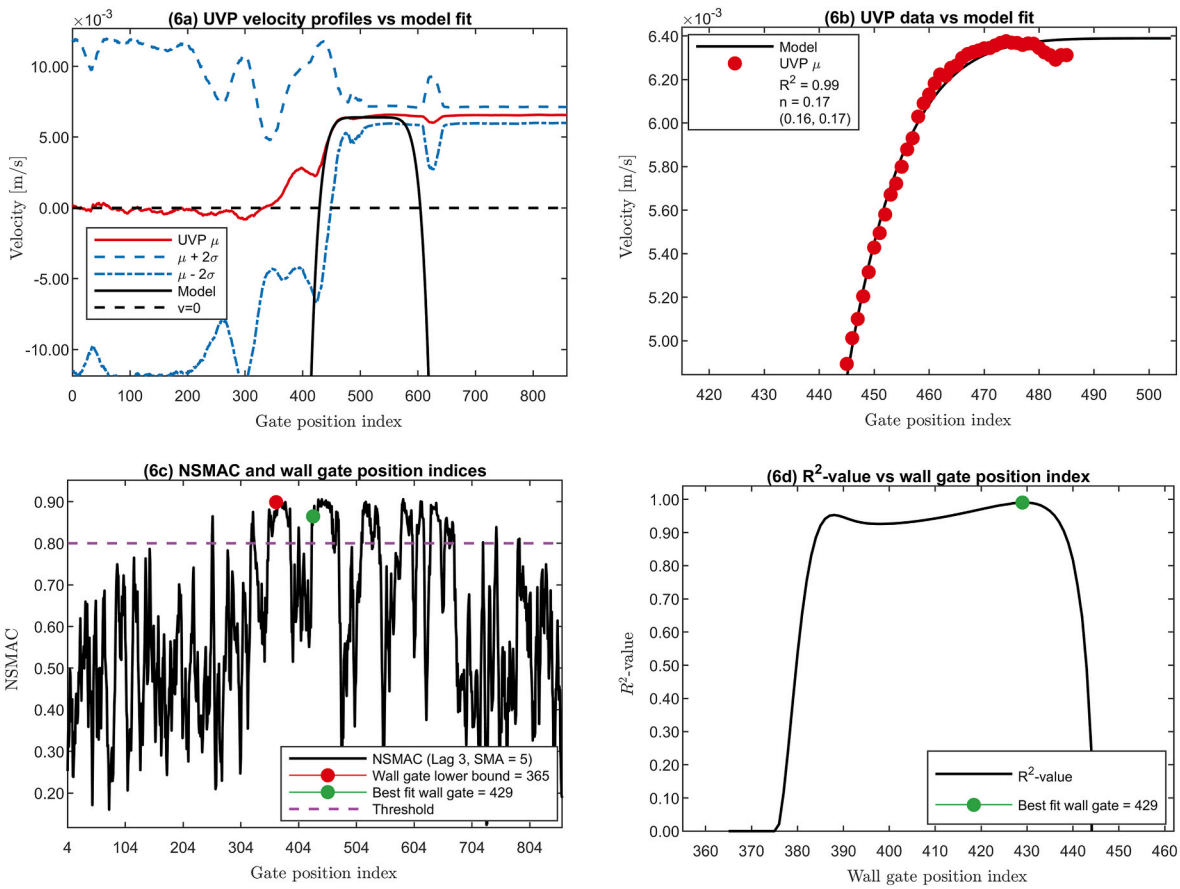

Fig. 6. (a) PUV velocity data with mean (μ), standard deviation (σ), the fitted model and zero velocity line for protein melt at 64% MC. (b) PUV mean data, fitted model near the velocity maximum, R^2-value, flow behavior index and associated 95% confidence interval. (c) NSMAC curve with the wall gate lower bound index, threshold value and best fit wall gate index. (d) R^2-value and best fit wall gate index.

melt flow, the methodology instead appears to be semi-quantitative due to the discrepancies between conventional rheometry and PUV data, possibly arising from the different temperature treatments, although the melts were assumed to be reversible. Since the velocity data was fitted in the die center region with limited fibre formation, the effect was still limited. Further, since there is apparent MC loss after extrusion, the differences between flow behavior indices obtained from PUV and conventional rheometry measurements are not surprising, since a higher MC should likely yield a higher value of the flow behavior index. On the other hand, this was not seen when only comparing the conventional rheometry measurements alone and the PUV measurements alone for the different protein melts. Nonetheless, it should here be emphasized that the temperature and associated shear history of the samples in the two different methods also are different, as described earlier.

5.2. Signal analysis and limitations

A limitation with the present signal analysis is the somewhat ambiguous range of relatively high integrity PUV data around the maximum velocity that should be used in the model fitting procedure. For each case, the data needs to be manually assessed in terms of the overall model fit characteristics and the SNR. Future development should focus on automating the selection of PUV data for the model fit based on a robust selection score that needs to be further investigated. The score needs to consider both the SNR close to the wall and the signal scattering in the fluid. Additionally, it is important to not only evaluate the fitted parameters based on one criterion, such as the R^2-value, in the present case. Instead, the landscape of the R^2-value over the relevant wall gate position indices in combination with the fitted velocity profiles and NSMAC curve can be used jointly for an overall assessment. It is further evident that the parameters defining the NSMAC curve introduce a trade-off between information loss and the allowable amount of correlation fluctuations when analyzing the PUV signals, where excessive smoothing or an improper threshold value may affect the wall gate position index estimate negatively. A potential solution may be found by e.g. engineering features out of the presented data for training of an AI algorithm to estimate both wall position and the data range to be used in the model fitting procedure.

5.3. Detecting fibre formation

The in-line PUV methodology presented in the present study can potentially be improved to detect fibre formation during HME extrusion by quantifying the local changes in relevant rheometric properties along the cooling die. However, this would require an improved transducer working at higher frequency with fewer cycles per pulse and better beam focusing that should generate less overlapping in the near wall region, thus providing better accuracy in the obtained velocity profiles for comparison with flow simulations. The cooling die also needs to be modified to enable the required position shift of the transducer as well as multiple local PD measurements along the cooling die, since the rheometric properties are expected to change continuously during fibre formation.

6. Conclusions

The present study has shown the potential to apply PUV methodology to estimate the flow behavior of glycerol, CMC and two pea protein

melts at different moisture content and at significantly different flow rates in an extruder die. Although the methodology is to be regarded as semi-quantitative at this stage for unstable low flow rates, future refinement of the methodology could potentially allow for non-invasive process control during extrusion of HMMA through determination of the instantaneous flow profile and local rheology of protein melts in the cooling die using combined PUV + PD methodology. Such information may also be of value to understand the fibre formation process in the cooling die. For future work, an improved transducer that provides better accuracy in the obtained velocity profiles, and a cooling die that allows for monitoring the flow profile along the whole cooling die, needs to be considered. Fortunately, for production scale extruders the larger die dimensions and, consequently, higher flow rates to obtain extrusion relevant shear rates are expected to facilitate the measurement process.

CRediT authorship contribution statement

Erik Kaunisto: Writing – original draft, Validation, Methodology, Funding acquisition, Formal analysis, Data curation. **Bahiru Tsegaye:** Writing – original draft, Investigation, Data curation. **Reinhardt Kotzé:** Writing – original draft, Software, Methodology. **Johan Wiklund:** Writing – original draft, Methodology. **Roland Kádár:** Writing – original draft, Methodology. **Mats Stading:** Writing – original draft, Supervision, Methodology, Investigation, Funding acquisition.

Declaration of competing interest

Erik Kaunisto, Bahiru Tsegaye and Roland Kádár have no individual financial or personal gains from conducting and publishing the present original research. Johan Wiklund, Reinhardt Kotzé and Mats Stading are all co-founders of Incipientus Ultrasound Flow Technologies AB and all have stocks in the company.

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Data availability

The data that has been used is confidential.

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