Monitoring of fat crystallization process using UVP-PD technique

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The response of the cocoa butter shear crystallization process to a step change in flow rate and temperature was investigated by measuring the instantaneous pulsed ultrasound Doppler based velocity profile (UVP) technique and pressure drop (PD) in a pipe section. In addition, the velocity of sound, attenuated amplitude of the transmitted signal and temperature were recorded. The temporal variation in rheological properties such as the shear rate dependent viscosity and the central plug radius in the pipe were determined by fitting the velocity profile and pressure drop to Power-law model. The linear dependence of sound velocity on the solid fat content in the cocoa butter crystal suspension previously determined using Nuclear Magnetic Resonance (NMR) technique was used to characterize solidification or melting behaviour. A new software developed was used to integrate on-line measurement of flow profiles, pressure difference, temperature, velocity of sound and the attenuated amplitude of the transmitted signal. The software also calculates velocity profiles form demodulated echo amplitude using Fast Fourier Transformation (FFT), determines the rheological properties, and provides a graphical user interface and tools for data visualization. The cocoa butter suspension was found to be shear thinning depending on the solid fat content and it was demonstrated that the cocoa butter shear crystallization process can be monitored and controlled using the UVP-PD methodology.

Keywords: Flow profiling, in-line rheometry, suspensions, fat crystallization, velocity of sound

1 INTRODUCTION

A novel method for in-line rheometry involving the measurement of an Ultrasound based Velocity Profile (UVP) and Pressure Difference (PD) in a pipe section was developed and tested at our laboratory for the flow of a wide variety of opaque model and industrial suspension systems such as chocolate, fat, shampoo and cellulose fibres in water suspensions. The in-line rheological results were compared with those measured by conventional off-line rheometers [1–5].

A shear crystallization process developed at our lab [6], which is meanwhile applied in the chocolate industry, was chosen to demonstrate the UVP-PD in-line rheometry measurement methodology. As it was suitable for the fat crystallization process, the UVP-PD technique is combined with measurements of velocity of sound, attenuation and temperature [7]. This allows an in-depth in-line investigation of the temporal behaviour of the cocoa butter crystal suspension in the process.

2 MATERIALS AND METHODS

2.1 Ultrasound transceiver and adapter cell

Transducers with 4 MHz and 5 mm active diameter from Imasonic (Besançon France) were used. The pipe adapter made from hard PVC allows positioning two transducers opposing each other to enable the simultaneous measurement of flow velocity profiles, velocity of sound and attenuation (at the given frequency)[7]. The Doppler angle is 30°. The transducers are in direct contact with the liquid but pulled back from the actual pipe wall so that the measurement starts at the end of the near field at the focal point of the ultrasound beam. In order to avoid the accumulation of air in front of the transducer, the whole adapter is fixed in a box and submerged in cocoa butter, which also provides a temperature control of the adapter.

2.2 Data acquisition hardware and software

A UVP-Duo instrument from Met-Flow (Lausanne, Switzerland) with a modified firmware that allows the access to the demodulated echo amplitude (DMEA) was used for the flow profile measurements. The two Kulite (Leonia, NJ, USA) pressure transducers and the thermocouples were connected using a National Instruments (Austin, TX, USA) FieldPoint. Velocity of sound (time of flight) and throughput amplitude were measured using a Yokogawa (Tokyo, Japan) DL 1520 oscilloscope.

The data acquisition from those entire instruments was controlled from software written with MATLAB (MathWorks, Natick, MA, USA). This software had a modular structure to allow a quick adaptation to different data acquisition hardware. Data from the UVP can be acquired using the Met-Flow ActiveX library either as DMEA or profile data as calculated with a time domain algorithm on the DSP in the UVP. Besides on-line data acquisition it is also possible to read files stored in various formats containing measurement data.

If the flow profile data is acquired as DMEA, the FFT algorithm is used to calculate the Doppler shift fre-

quencies. Besides data acquisition and signal processing, the software also provides a graphical user interface to set the various measurement parameters and tools for the visualization of the data.

2.3 UVP-PD method

The UVP-PD method allows the estimation of rheological parameters for Newtonian and Non-Newtonian liquids or suspension by combining profile measurement, pressure difference and a flow model such as Power-Law ($\tau = K\dot{\gamma}^n$). Details on the technique can be found elsewhere [1-5]. The fitting procedure is also an integral part of the software described in the previous section.

2.4 Cocoa butter fat crystal suspension

Cocoa butter used for chocolate production is a mixture of triglycerides with a polymorphic crystallization behaviour that has a melting temperature around 32 °C. The crystal content is increased up to 25% during the shear crystallization developed at our laboratory [6].

2.5 Flow loop and crystallization process

The fully temperature controlled flow loop shown in figure 1, discussed in detail in [6], consists of a stirred cocoa butter feed tank, a two stage shear crystallizer and the measurement pipe section. The two pressure sensors are 1 m apart from each other.

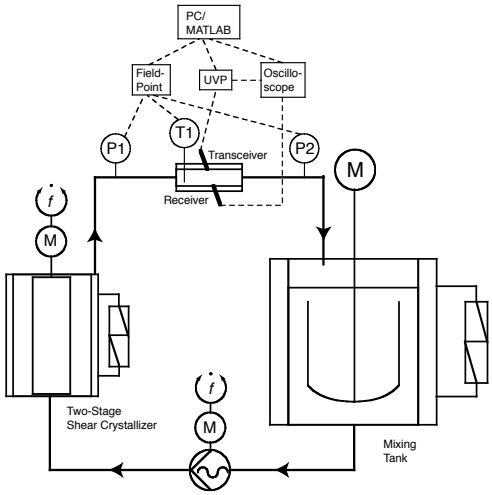

Figure 1 Scheme of the crystallizer flow loop, its instrumentation and data acquisition devices. P1, P2: pressure transducers; T1: thermocouple; M: motor; f: frequency transformer.

3 RESULTS

Normally the shear crystallizer operates at steady-state at a given flow rate of the cocoa butter crystal suspension with a constant concentration of the cocoa butter crystals. In order to investigate the changes in the rheological behaviour with time due to changes in the process operating variables, a set of experiments were carried out as described below.

Figure 2 shows an overview of the time dependent variation in the parameters measured over 2 h due to the following three phases of variations in process conditions starting with liquid cocoa butter flowing at steady-state with a throughput of 19.3 kg/h at 43 °C.

3.1 Crystallization process parameters

(1) The two-stage shear crystallizer unit was started when the flow loop was operating at a cocoa butter throughput of 19.3 kg/h. The first and second stages of the shear crystallizer unit were cooled to 15 °C and 27 °C respectively. The head and spindle of the shear crystallizer, and the pipes were cooled to 32 °C. (2) At 2500 s, the cocoa butter crystal suspension flow rate was reduced to 11.3 kg/h, which resulted in increase in the solid fat content up to about 20–25 %. The temperatures of the first and second stages of the crystallizer are the same as in phase 1. (3) At 8430 s, the cocoa butter crystal suspension was melted by switching off the shear crystallizer and heating the pipes to 43 °C resulting in the flow of liquid cocoa butter at a flow rate of 11.3 kg/h.

3.2 Measured parameters

The measured parameters can be broadly classified into four categories: (i) the process parameters such as the pressures and temperatures at the inlet and outlet of a pipe section after the shear crystallizer, (ii) the acoustic parameters such as the sound velocity and the amplitude of the transmitted ultrasound signal through the cocoa butter suspension, (iii) the flow velocity profile along the pipe diameter, and (iv) the rheological parameters such as the shear rate, shear stress, apparent viscosity determined based on the fitted Power-law model parameters *n* and *K* obtained using the velocity profile and pressure drop in pipe section.

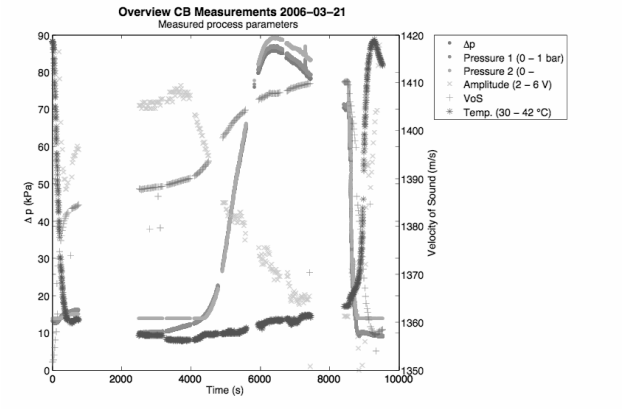

Figure 2: Overview for all the measured values of the crystallization process.

In the first phase (for less than 2500 s), it can be seen from Figure 2 that the temperature decreased from 41 °C to 32 °C while the velocity of sound increased from 1352 m/s to 1384 m/s.

In the second phase (between 2500 s and 8430 s) involving reduction of flow rate to 11.3 kg/h, the concentration of cocoa butter crystals (referred as solid fat content SFC) increased. This resulted in an increase in the sound velocity from 1390 to 1410 m/s and a steep rise in the pressure drop from 10 to 90 kPa. The SFC was not directly measured but under similar process conditions a value of around 20 % was reached in this flow loop. The attenuation increased with the increase of the SFC, thus the amplitude of the signal received with the second transducer was reduced by a factor of two from 5.5 to 2.75 V. The calibration of the amplifier of the sensor at the pipe upstream was not optimally adjusted, thus the pressure difference until about 4300 s was not exact, which could influence the value of *K*.

In the third phase of the process condition variation (later than 8430s) involving switching off the shear crystallizer and heating of the pipe, the temperature, and the amplitude of ultrasound signal received increased until their values corresponded to those for the liquid cocoa butter at about 43 °C. The pressure drop and the velocity of sound also decreased until becoming equal to those for the liquid cocoa butter.

3.3 Calculated results

Figure 3 shows the development of the root mean square of the DMEA (normal) over the whole measurement. The echo intensity increased during the first four measurement blocks after the flow rate reduction (between profiles 75 and 475) in region near the transducer. After profile no. 450 the intensity in the first 10 channels dropped suddenly from the maximum to the minimum.

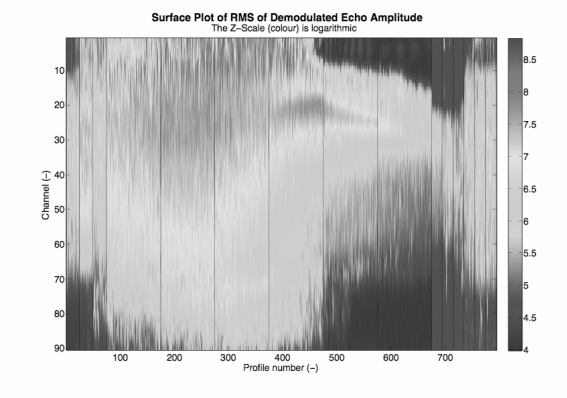

Figure 3: Surface plot of the root mean square of the demodulated echo amplitude with a logarithmic scale in Z (Color) direction. The measurement blocks are marked with vertical lines.

The drop in pressure coincided with the drop of the demodulated echo amplitude. At the same time the slope of the velocity of sound became nearly zero. The pressure continued to drop subsequently while the velocity of sound continued to increase at a slower rate than before.

Figure 4, 5 and 6 show the development of the flow velocity profiles during the process. The first three measurement blocks (before profile no. 100) were measured with a higher flow rate, therefore the drop afterwards. Then, during the next six measurement blocks, until after profile no. 600 the crystallization took place. The minimum velocity was reached at the end of the forth measurement block before profile no. 500. An interesting point was the non-zero velocities that were measured from this point onwards in front of the transducer (the actual pipe starts only around channel 15). This was probably caused by a lack of signal (see Figure 3). This artefact disappeared when the cocoa butter became mostly liquid in the last three blocks.

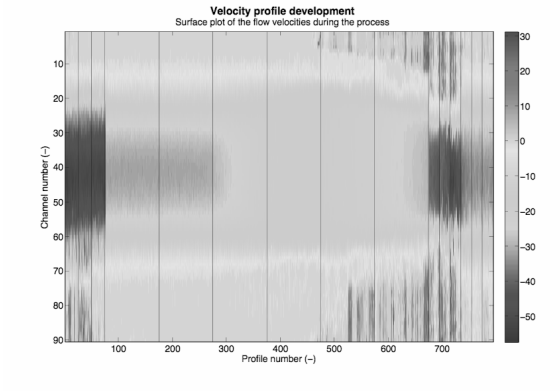

Figure 4: Surface plot of the flow velocities during the crystallization process. The development of velocity profiles along the diameter of the pipe is shown in Figures 6.64 and 6.65 during the three phases of the variation in process conditions.

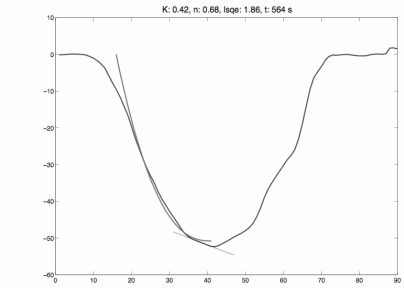

Figure 5: Measured (full curve) and fitted (half curve) velocity profiles after 564 s (liquid cocoa butter).

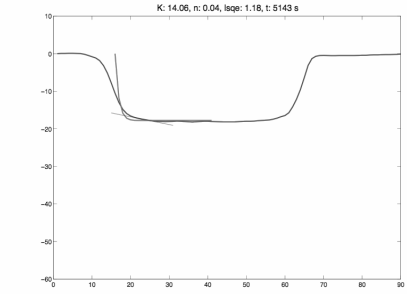

Figure 6: Measured (full curve) and fitted (half curve) velocity profiles after 5143 s (crystal suspension).

Figure 7 shows the values of power-law rheological model ($\tau = K\dot{\gamma}^n$) parameters *n* and *K* determined by fitting the velocity profile equation. Initially, the cocoa butter was in liquid state so that *n* is equal to 1 as expected for Newtonian liquids. Then, in the first phase of the crystallization (until 1000 s) the value of *n* drops quickly (corresponding to the temperature reduction) to a value around 0.6. In the first phase after the reduction of the flow rate (after 2500 s) the values of *n* and *K* change slowly. This is due to the time of approximately 20 min it takes for the passage from the vessel through the shear crystallizer to the profile measurement. Then after 4000 s there is a kink in the value of *K*, this is probably due to the problem with the lower threshold of the measurement of the upstream pressure. So before this point the real pressure difference is smaller than the measured one, and thus the calculated value of *K* is too high and goes down to the real value when the pressure difference is actually measured after 4500 s. The value of *n* is independent of the pressure difference.

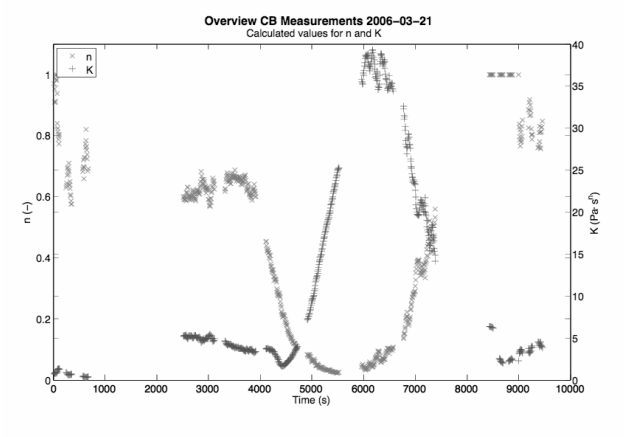

Figure 7: Development of *K* and *n* during the crystallization process.

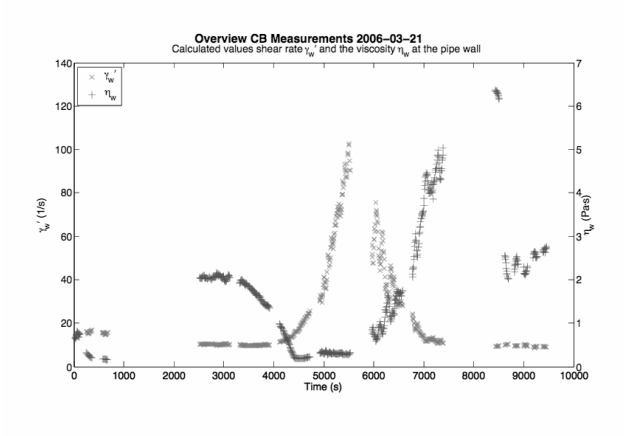

Figure 8: Calculated shear rates ($\dot{\gamma}_w$) and viscosity (η_w) at the wall.

At the pipe wall we find the highest shear rates and thus (shear thinning character of the crystal suspension) the lowest viscosities. Figure 8 shows the estimate of the corresponding values calculated from the fitted velocity profile with $\dot{\gamma}_w = \left(R\Delta P/2LK\right)^{1/n}$ and $\eta_w = \tau_w/\dot{\gamma}_w = K\left(R\Delta P/2LK\right)^{1-1/n}$.

The wall viscosity begins dropping already after 3000 s while the apparent shear rate remains constant until 4000 s. This is due to the difference in the exponent. For $\dot{\gamma}_w$ it is $1/n$ while for η_w it is $1-1/n$.

The radius of the region in the middle of the pipe where the velocity profile is virtually flat, is estimated by analysing the derivative of the profile. This radius r_p and the corresponding shear stress $\tau_p = r_p \Delta P/2L$ are shown in Figure 9. The distribution of r_p is discrete because its possible values correspond to the measurement channels.

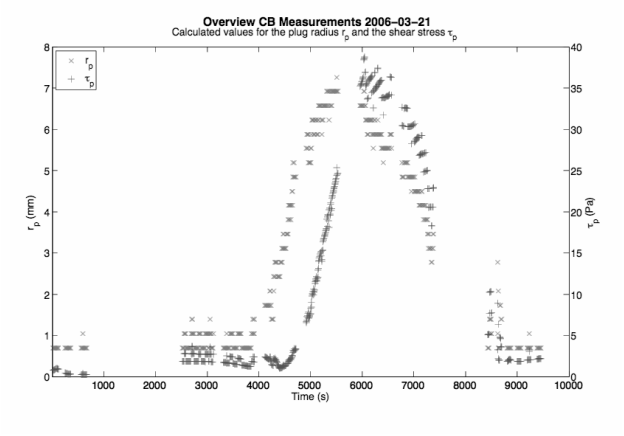

Figure 9: τ_p and the estimated radius of the plug r_p.

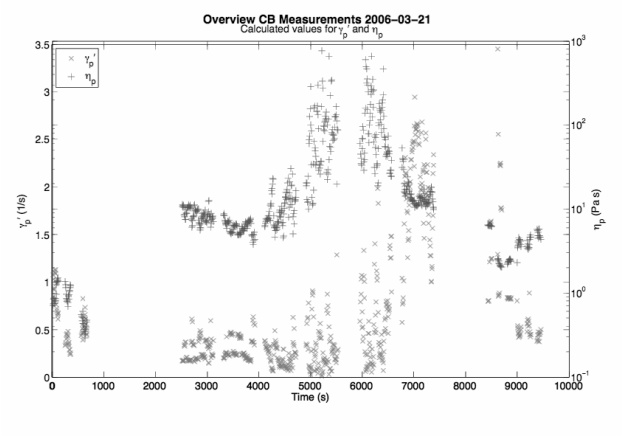

Figure 10: Calculated shear rates $\dot{\gamma}_p$ and viscosity η_p for the plug.

Figure 10 shows $\dot{\gamma}_p = \dot{\gamma}_w\left(r_p/R\right)^{1/n}$ and $\eta_p = \tau_p/\dot{\gamma}_p$. As discussed earlier, the value of *n* decreased with time as the crystal concentration increased resulting in an increase in pressure drop. This implied that the crystal suspension became

more shear thinning gradually. At any instant of time, the shear rate increased along the radius of the pipe being maximum at the pipe wall and minimum at the plug radius. Consequently, the viscosity is minimum at the wall of the pipe while it is maximum at the radius of the plug. As time increased, the radius of the plug increased so that the corresponding viscosity also increased resulting in increased pressure drop.

3.4 Limitations of the current setup

In order to avoid measurements in the near field of the ultrasound beam the transducer were pulled back 17 mm. As for this experiment the pipe diameter is 16 mm and the diameter of the transducer housing is 8 mm there are three major drawbacks: (i) the actual flow field is influenced by the geometry of the adapter and (ii) especially with the crystal suspension there is the disadvantage of possible crystallization in the cavities in front of the two transducers where there is no flow. Because the transducer adapter block is made from Hard PVC it is also not possible to control the temperature precise and fast enough. (iii) In the presented measurement both transducers were pulled back 17 mm, thus two third of the velocity of sound measurement are not inside the actual pipe diameter. Consequently, sound velocity and throughput signal amplitude are not fully representative for the situation in pipe flow. (iv) Since average sound velocity is measured, its precise value between transducer front and the start of the pipe radius is not determinable. Due to the assumed crystallization in front of the transducer, the velocity of sound is not distributed evenly between the two transducers and thus the distances are not determinable precisely.

A new transducer type that is under development should solve these limitations. This transducer features a so called «delay line» that is fixed to the transducer front and is in flush with the pipe wall. So the delay line contains the near field and at the same time focuses the beam inside the pipe so that the divergence angle is minimized and thus the measurement would be more precise.

4 CONCLUSIONS

It was shown that the UVP-PD technique combined with the measurements of velocity of sound and attenuation is an interesting approach for the in-line monitoring of a fat crystallization process. It is possible to monitor crystal content (by the velocity of sound) and the flow behaviour simultaneously. By adapting the transducer to the specific pipe diameter and adapting the UVP electronics for the specific needs it should be possible to implement an automatic in-line process control system for the cocoa butter shear crystallization and other similar applications.

5 ACKNOWLEDGEMENTS

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NOMENCLATURE

K	Pa s^n	Flow consistency index
L	m	Length of pipe for pressure drop
n	–	Flow behaviour index
ΔP	Pa	Pressure difference
r	m	Radius
R	m	Pipe radius
$\dot{\gamma}$	1/s	Shear rate
η	Pa s	Viscosity
τ	1/s	Shear stress

Indices:

p	Plug
w	Wall

REFERENCES

[1] Ouriev B: Ultrasound Doppler based in-line rheometry of highly concentrated suspensions, Ph.D. Dissertation ETH No. 13523, 2000, Swiss Federal Institute of Technology, Zurich.

[2] Ouriev, B, et al: Industrial application of UVP-PD technique: Steady shear pipe flow of chocolate suspension in pre-crystallisation, Review of Scientific Instruments 74 (2003), 5255-5259.

[3] Ouriev B, Windhab E: Transient flow of highly concentrated suspensions investigated using UVP-PD method, Measurement, Science and Technology 14 (2003), 1963-1972.

[4] Ouriev, B, Windhab E: Novel ultrasound based time averaged flow mapping method for die entry visualization in flow of highly concentrated shear- thinning and shearthickening suspensions, Measurement, Science and Technology Journal 14 (2003), 140-148.

[5] Wiklund, J, et al.: 3rd-International Symposium on Ultrasonic Doppler Methods for Fluid Mechanics and Fluid Engineering, 2002, 69-76, EPF Lausanne.

[6] Zeng Y: Impf- und Scherkristallisation von Schokoladen, Ph.D. Dissertation ETH No. 13798, 2000, Swiss Federal Institute of Technology, Zurich.

[7] Birkhofer B, et al.: 4th International Symposium on Ultrasonic Doppler Method for Fluid Mechanics and Fluid Engineering, 2004, 65-68, Sapporo.