# In-line ultrasound based rheology of concentrated suspensions

Johan Wiklund[1] a and Mats Stading[1*]

[1]SIK, The Swedish Institute for Food and Biotechnology, SE-402 29 Göteborg, Sweden
*To whom correspondence should be addressed
E-mail address: mats.stading@sik.se

ABSTRACT

A new rheological method that involves simultaneous measurement of radial velocity profiles, using a pulse Doppler ultrasound velocity profiling technique (UVP), in combination with a pressure difference technique (PD) was used in this work to determine rheological properties directly in a process line. Complex non-Newtonian industrial suspensions were investigated.

## INTRODUCTION

The ability of the processing industry to develop innovative products largely depends on the ability to control the manufacturing process. Foods serve as good examples and are very complicated materials as they are often inhomogeneous, non-Newtonian, highly concentrated and non-transparent fluids during processing. The control of temperature and mass flow is well developed whereas there is insignificant control of rheological properties, especially within the process line.

The rheological behavior has so far been analyzed using commercially available instruments, such as conventional rotational rheometers. Most of these commercially available instruments are usually not suitable for in-line rheological measurements and quality control as they are based on invasive methods, and have geometries that can affect and disturb the flow. Real flow situations can almost never be obtained using these instruments and they may also cause severe bio-safety problems due to contamination, which also limits their applicability. Without the introduction of new in-line methods, real-time control of the process and guaranteed product quality are unlikely to be achieved in the modern factory.

In case the velocity profile can be determined, preferably in-line, the rheologically important flow curve, i.e. shear viscosity function etc., can be derived directly from a non-linear model fit of the velocity profile and corresponding pressure drop data. The UVP-PD methodology complies with these requirements and constitutes perhaps the most promising techniques for a new in-line/on-line rheometric concept.

### UVP technique

Ultrasound based methods are currently used to measure velocity profiles in diverse systems ranging from blood to complex industrial systems such as pulp suspensions. Takeda[1], employed and modified a pulsed ultrasound echo graphic technique, which had been originally developed for the measurement of velocity profiles of blood in human arteries, to flowing suspensions of more general fluids.

Because of the pioneering work of Takeda this technique has been established for measuring velocity profiles of fluid flow in both physics and engineering and is now manufactured as an Ultrasound Velocity

Profile (UVP) Monitor by the company Met-Flow SA, Switzerland.

The instrument is based on the combination of a pulsed ultrasound echography and Doppler shift method. The emitted pulses are echoed back towards the transducer by reflection surfaces moving with the flowing suspension. Instantaneous spatial velocity distributions along the axis of an emitted pulse beam are thus obtained by measuring the Doppler shift in the frequency of the reflected ultrasound and time delay. Technical details are given in[1].

UVP-PD method

The main principles of the in-line UVP-PD methodology has recently been proposed and described in literature by several groups e.g. Brunn et al.[2], Wunderlich et al.[5], Ouriev[6], and Wiklund.[9,11].

According to recent literature, the in-line UVP-PD method has been tested with great success for a number of fluid systems both Newtonian and non-Newtonian. Some analyzed systems exhibit very complex flow characteristics such as surfactants, highly concentrated food systems/suspensions and tomato/paper pulp. Detailed information about these experiments and results is given e.g. in Muller et al.[3], Shekarriz et al.[4], Ouriev[6], Dogan et al.[8] and Wiklund et al.[7, 9-11].

In this study, the in-line UVP-PD methodology was tested with success on highly concentrated suspensions such as cellulose pulp. The complex rheology of a fish soup containing both a fat emulsion base with spices and large (~2cm) fish, squid and mussels pieces was also studied in a flow loop, under almost actual processing conditions.

EXPERIMENTAL SET-UP

Two experimental flow loops, have been used. The first loop and experimental procedure for the pulp suspensions is described in detail in[10] and the second one in[11]. Some details about the second loop are given here. Ultrasonic velocity profiling was performed using a UVP-Duo Model Monitor, from Met-Flow SA, Switzerland. The equipment was controlled using a master PC with novel software. A digital 4 channel oscilloscope was used to monitor the UVP signals.

The second flow loop consists of a stainless steel tanks with agitators, a positive displacement pump to re-circulate the investigated suspension, stainless steel piping with an inner diameter of 23.5 or 35.5 mm respectively. A flow adapter, shown in Figure 1, made of a plastic composite or stainless steel fitted with 2-8 MHz ultrasound transducers that enables non-invasive measurements constitutes the measurement section for velocity profiling. Three pressure sensors for pressure drop measurements and several temperature transmitters, mass and volumetric flow meters were also used. A master PC was used for all data acquisition and post processing of UVP data in Matlab using novel software.

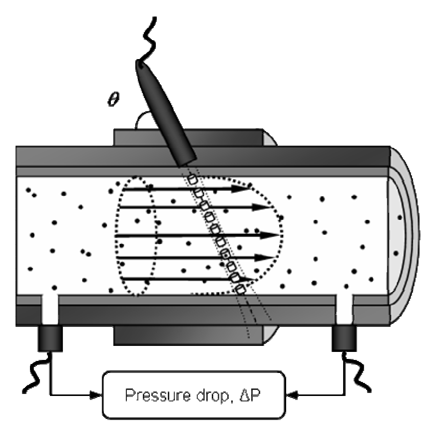


Figure 1. Schematic of the flow adaptor with one ultrasound transducer and two pressure sensors.

EXPERIMENTAL PROCEDURE

The tank was filled with the suspension, agitated mildly, the pump was then set to the desired flow rate and the flow was allowed to attain steady-state. The sound velocity in the suspensions was measured at different temperatures in-line using the method described in[11]. UVP data acquisition was in the order of few milliseconds per profile. Velocity profiles were recorded and validated simultaneously as pressure drop, flow rate and temperature measurements for each flow rate.

In order to obtain rheological data, such as the generalized power-law exponent $n$ and consistency index $K$, the UVP-PD method described, e.g. in[11] was applied to the recorded data. Rheological parameters were derived from non-linear regression analysis using e.g. Herschel-Bulkley model (pulp) and power-law (fish soup) model given by Eq. 1.

$$\tau = K\gamma^n \qquad (1)$$

where $\tau$ is the shear stress, $K$ is the consistency index and $n$ is the flow exponent (describing the shape of the profile). Using the integrated form of this model, the radial velocity profile will be given by Eq. 2.

$$v(r) = \left(\frac{\Delta P}{2LK}\right)^{\frac{1}{n}} \frac{R^{1+\frac{1}{n}}}{1+\frac{1}{n}} \left(1 - \left(\frac{r}{R}\right)^{1+\frac{1}{n}}\right) \qquad (2)$$

where $r$ and $R$ is the inner and outer pipe radius respectively and $L$ is the distance between the pressure transducers.

The obtained velocity profiles and pressure drop data were then used in the non-linear fitting method used for evaluating the rheological parameters $n$ and $K$. Velocity data in the range from $r = 0$ to $R$ were used when evaluating these parameters. The volumetric flow rate was obtained from integration of the velocity profiles.

The fish, squid and mussels pieces were removed from the samples of fish soup in order to be able to perform measurements in a conventional rotational off-line rheometer, Bohlin VOR. The samples were analyzed over a shear rate range that corresponded to those in the flow loop and the power-law parameters $n$ and $K$ were then finally determined.

RESULTS AND DISCUSSION

The presented results are chosen to give an overview of the possibilities with the UVP-PD method as well as the limitations.

Figure 2 shows that it was possible to obtain instantaneous and complete velocity profiles using the UVP technique in only a couple of milliseconds in such a difficult system as a fish soup containing large (~2cm) fish, squid and mussels pieces. In addition, Figure 2 also shows that the shape of the velocity profiles gradually changed over time from flat plug-flow behaviour into more Newtonian behaviour with increasing flow rate and more pronounced grinding of the solid pieces.

It was also noted that a decrease in penetration depth towards the far side from the transducer resulted in loss of velocity gradient information. This is visible as non-zero velocities on left side in the figure as measurements were performed from right to left using a single transducer.

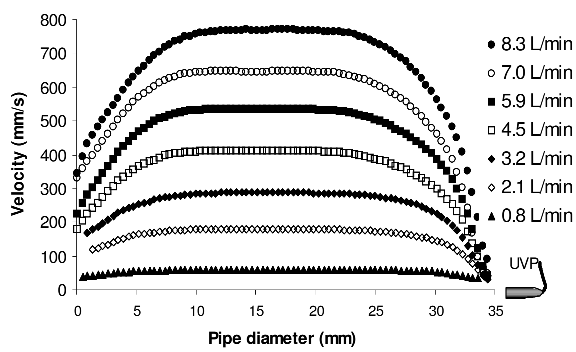


Figure 2. Average velocity profiles for a fish soup containing large pieces (~2cm) of fish and mussels

The power-law parameters $n$ and $K$, obtained using the UVP-PD methodology, is listed in table 1 on the following page.

Table 1. Power-law fitted parameters from UVP-PD measurements (in-line).

| Pressure drop (Pa) | Flow rate (L/min) | K | n |
|---|---|---|---|
| 9900 | 0.8 | 20.0 | 0.21 |
| 13900 | 2.1 | 19.5 | 0.25 |
| 15400 | 3.2 | 18.2 | 0.27 |
| 18300 | 4.5 | 16.2 | 0.32 |
| 18500 | 5.9 | 14.0 | 0.33 |
| 16700 | 7.0 | 12.1 | 0.33 |
| 17700 | 8.3 | 11.8 | 0.34 |

As shown in Table 1, it was possible to follow the gradual change in rheological behavior of the fish soup (in-line) as the power-law parameters gradually changed with increasing flow rate. This corresponds to a gradually increasing grinding of the fish soup, as the fish, squid and mussels' pieces were more and more torn apart into smaller pieces with time and increasing flow rate, i.e. at increasing shear rates.

Table 2. Power-law fitted parameters from Bohlin VOR-rheometer measurements (off-line).

| Test No. | K | n |
|---|---|---|
| 1 | 8.3 | 0.45 |
| 2 | 7.9 | 0.46 |
| 3 | 7.4 | 0.47 |

It can be seen in Table 2 above that the power-law parameters obtained using a conventional off-line rheometer differed from those in Table1, obtained using the UVP-PD method. Although the samples were analyzed over a shear rate range that corresponded to the volumetric flow rates (or i.e. corresponding shear rates) in the loop, the results from the two methods differed.

The flow index $n$ was in this case higher, implicating a more Newtonian behavior, but also with a lower $K$, implicating a lower viscosity compared to the UVP-PD results. The discrepancy between the two methods was most likely caused by the fact that the fish, squid and mussels pieces were removed from the fish soup samples in order to be able to perform measurements in the conventional rotational off-line rheometer. This clearly shows that the off-line technique in this case measures on a modified system under unrealistic flow a condition that does not correspond to the actual ones in the process. In addition, this technique also only produces point-wise data which made it impossible to study the gradual change in rheological behavior.

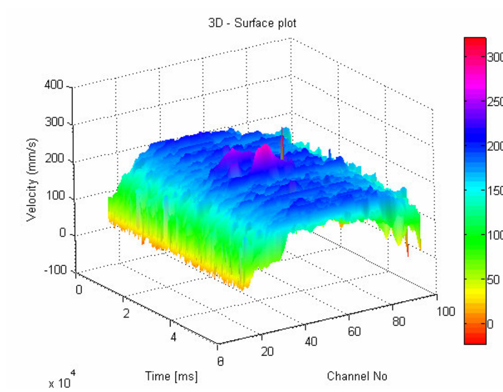


Figure 3. 3D-surface plot of the fish soup showing the velocity profile evolve over time.

Results further showed that it was possible to obtain information on the local position of the fish pieces in the flow field from a 3D-time plot of the velocity profiles when those were compared with digital video recording of the flow. The local position of the individual fish, squid and mussels pieces in the fish soup could be detected, as shown in Figure 3 above, as big lumps travelling with higher velocities in specific parts of the velocity profiles. Detailed information about these experiments is given in Wiklund et al.[12].

For the first time, non-invasive velocity profile measurements in highly concentrated pulp suspensions of up to 7.8 %(w/w) have been performed simultaneously using the UVP and LDA techniques in an experimental pipe flow loop. Results from the highest concentration are presented in this paper. Instantaneous radial velocity profiles in pulp suspensions at concentrations ranging from 0.74 %(w/w) up to 7.8 %(w/w) have been obtained non-invasively through a 5 mm thick Polymethyl methacrylate (PMMA), measurement section using the UVP technique. Figure 4 shows the velocity profile data for the highest concentration of 7.8 %(w/w).

Our results showed that both techniques can be used with good agreement in terms of absolute velocities in much more concentrated pulp suspensions than what has been reported so far in the literature.

Furthermore, results show that the UVP technique could be optimized in such way that velocity gradient information close to the pipe wall could be obtained. The penetration depth was also sustained which enabled almost complete velocity profile information even for the highly concentrated 7.8 %(w/w) pulp suspension.

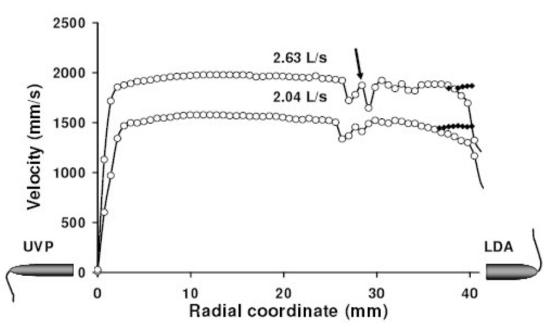


Figure 4. UVP (circles) and LDA (diamonds) data for 7.8 %(w/w) pulp suspension at 2.04L/s and 2.63L/s

A decrease in penetration depth due to ultrasound attenuation, multiple echoes (as indicated by e.g. an arrow in Figure 4) and the effect of various disturbing measurement effects, such as the gain settings used, was found for this system. These effects e.g. resulted in constant plug behavior of the velocity profile, in which the velocity gradient information is lost, towards the far end pipe wall (right) from the UVP transducer.

As shown in figure 4, results further show that the LDA technique works even in strongly opaque systems like a 7.8 %(w/w) pulp suspension with a sustained penetration depth of up to several millimeters. No addition of seeding particles was needed as the pulp fibres were found to work sufficiently for both LDA and UVP techniques.

Combined with simultaneous pressure drop measurements, it was found that the UVP technique can provide rheological information in-line for complex pulp suspensions. Detailed information about these experiments is given in Wiklund et al.[10].

## CONCLUSIONS

Results from this study showed that the UVP-PD method can be applied for non-invasive, rheological measurements in-line, of complex, non-transparent, fish soup containing both a fat emulsion base with spices as well as fish, squid and mussels pieces. In addition, the gradual change from non-Newtonian to more Newtonian behavior could be monitored in real-time, in-line. Experimental results showed a discrepancy with off-line measurements using conventional rotational rheometers as the latter were performed on a rheologically different system (no pieces) and under different flow conditions.

Results from the pulp study showed that both LDA and UVP techniques can be used with good agreement to obtain accurate velocity profile data in much more concentrated pulp suspensions than what has been reported so far in the literature. Velocity profiles in pulp suspensions, ranging from 0.74 %(w/w) up to highly concentrated 7.8 %(w/w) were obtained using both LDA and UVP. The UVP-PD method can thus be regarded as a powerful in-line, as well as research tool for e.g. process control.

ACKNOWLEDGMENTS

This study was conducted as part of a PhD project which is financed by Tetra Pak Processing System AB and Vinnova, the Swedish Agency for Innovation Systems. Met-Flow SA, Switzerland is gratefully acknowledged for technical support on UVP technique.

REFERENCES

1. Takeda Y., (1986) "Velocity profile measurement by ultrasound Doppler shift method", *International Journal of Heat Fluid Flow*, **7**, pp. 313-318.

2. Brunn, P. O., J. Vorwerk, and R. Steger, (1993a), "Optical and acoustic rheometers: three examples," *Rheology*, **93**(3), 20-27.

3. Muller, M., P. O. Brunn, and T. Wunderlich, (1997), "New Rheometric Technique: the Gradient-Ultrasound Pulse Doppler Method," *Appl. Rheol.*, **7**, 204-210.

4. Shekarriz, A., and D. M. Sheen, (1998), "Slurry pipe flow measurements using tomographic ultrasonic velocimetry and densitometry", Proceedings of FEDSM'98, June 21-25,Washington, DC, USA, 1-8.

5. Wunderlich, T., and P. O. Brunn (1999), "Ultrasound pulse Doppler method as a viscometer for process monitoring", *Flow Meas. and Instr.*, **10**(4), 201-205.

6. Ouriev B., (2000), "Ultrasound Doppler based In-Line Rheometry of Highly Concentrated Suspensions", *ETH dissertation No. 13523*, ISBN 3-905609-11-8, Zurich, Switzerland

7. Wiklund, J., Johansson, M., Shaik, J., Fischer, P., Windhab E, Stading, M. and Hermansson, M.,(2002), "In-line ultrasound based rheometry of industrial and model Suspensions flowing through pipes", Trans. Third International Symposium on Ultrasonic Doppler Methods for Fluid Engineering, EFPL, Lausanne, Switzerland, 69-76.

8. Dogan, N., M. J. McCarthy, and R. L. Powell, (2002), "In-line measurement of rheological parameters and modelling of apparent wall slip in diced tomato suspensions using ultrasonics", *J. Food Sci.*, **67**(2), 2235-2240.

9. Wiklund, J. (2003), "Rheological In-line techniques based on ultrasound Doppler methods for the food industry. A literature survey," *SIK – Report SR-710, ISBN 91-7290-225-X, Göteborg, Sweden*

10. Wiklund J.A., Pettersson A.J., Rasmuson A. and Stading M. (2005), "A Comparative Study of UVP and LDA Techniques for Pulp Suspensions in Pipe Flow", *AIChE Journal, in press.*

11. Wiklund, J., M. Stading, (2005), "Ultrasound Pulsed Doppler and Pressure Difference based method for In-line Process Monitoring and Viscometry", *Manuscript in preparation*

12. Wiklund, J., M. Stading, (2005), "In-line UVP-PD based process viscometry of highly concentrated suspensions containing large particles", *Manuscript in preparation*