Ultrasound Doppler based In-line rheometry for processing applications

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## ABSTRACT

Liquid displacement of model and industrial fluids in pipes was investigated using the in-line ultrasound Doppler based UVP-PD method, combining Ultrasound Velocity Profiling and Pressure Difference The profile measurements. velocity development and changes in rheological properties during the fast transient displacement experiments were successfully monitored in real-time with 8-10 updates/second.

## INTRODUCTION

Liquid displacement of one fluid by another, miscible or immiscible is a very common and important process in fluid industry. Typical situation examples are start-up or shut-down of operations where e.g. a transition from water to product or vice versa takes place for intermediate rinsing in order to prepare a product change, or the use of the same filler for different products.

A mixing zone then appears between the displacing and displaced fluid because of convection and diffusion phenomena. Monitoring the displacement zone is very important in order to ensure product integrity and to reduce product loss, waste load and related costs.

Previous experiments have mainly focused on displacements in vertical pipes and capillaries and very little has been done on horizontal pipes. The ultrasound Doppler velocity profiling (UVP) technique is a highresolution method capable of measuring instantaneous velocity profiles in liquid flow along the pulsed ultrasonic beam axis. UVP is now accepted as an important tool for measuring flow profiles in research and engineering due to the pioneering work by Takeda<sup>1</sup>.

In an earlier study<sup>2</sup> we have shown that the UVP method was able to study the displacement of yoghurt by water, and that results were in good agreement with CFD simulations.

An in-line method for rheological characterization of opaque model and industrial fluids has been developed, extensively investigated, continuously improved and evaluated over a decade by research groups at SIK and ETH. The method is based on the UVP technique in combination with pressure drop (PD) measurements in a section of pipe. The method, known as UVP-PD, is described in detail by Wiklund et al.<sup>3,4</sup>.

The UVP-PD method has now been successfully applied to concentrated model suspensions<sup>5</sup>, to industrial fluids and large particulate suspensions<sup>6</sup> and to cellulose pulp<sup>7</sup>.

The aim of this study was to characterize the displacement, related mixing zone and changes in rheology when one fluid was displaced by another with different rheological properties using the UVP-PD method.

## MATERIALS AND METHODS

#### Model and industrial fluids

Four sets of model systems were used. They all exhibit power-law behaviour over the investigated range of shear rates and also adequately cover and represent different classes of fluids, all relevant for the food industry. In addition, two sets of industrially produced foods with verv similar characteristics in each set were used to test the limitations of the UVP-PD system. The model systems and industrial fluids are presented in Table 1 and more details are given in Wiklund et al.<sup>8</sup>.

Table 1: Model systems and industrial fluids

Fluid A	Fluid B	Characteristics	Miscibility
PEG400	Aqueous	Newtonian $\rightarrow$	miscible
	syrup	Newtonian	
PEG400	Sunflower	Newtonian $\rightarrow$	immiscible
	oil	Newtonian	
Xanthan	PEG400	Non-Newtonian $\rightarrow$	miscible
		Newtonian	
Xanthan	Sunflower	Non-Newtonian $\rightarrow$	immiscible
	oil	Newtonian	
Créme	Créme	Non-Newtonian	miscible
Fraiche 1	Fraiche 2	Different KA, KB	
		Similar n <sub>A</sub> , n <sub>B</sub>	
Yoghurt 1	Yoghurt 2	Non-Newtonian	miscible
		Similar K <sub>A</sub> , K <sub>B</sub>	
		Different n <sub>A</sub> , n <sub>B</sub>	

#### Off-line viscometry

A rheological characterization of the model systems was made off-line prior to the in-line experiments in a controlled stress rheometer. Rheologica Stresstech HR (Rheologica Instruments, Lund, Sweden). The industrial fluids were characterized using a RheoMat 180 (proRheo GmbH, Althengstett, Germany). The results from viscometric measurements the were compared at room temperature and over a shear rate range, 0.1-100 s<sup>-1</sup> that matched the conditions in the experimental loop.

## In-line viscometry and the UVP-PD method

The experimental flow loop with hardware, the UVP-PD method for pipe

viscometry and the constitutive equations used is presented in detail Wiklund et al.<sup>3, 4</sup> ,thus only a short summary is given here. A schematic illustration of the experimental flow loop is shown in Fig. 1.



Figure 1. Schematic illustration of the experimental flow loop and instrumentation.

The flow loop consists of a closed stainless steel pipe circulation system with an inner pipe diameter of 35.5 mm. A positive displacement pump (On-line OL2/0025/10, Johnson Pump, UK) was used to re-circulate the sample fluids. Two thermocouples and an electromagnetic volumetric flow meter (Discomag DMI 6531. Endress+Hauser AB. Bromma, Sweden) were used to monitor the flow rate and temperature. The pressure difference was measured over a distance of 2.52 m using two pressure sensors (ETP80, ABB Automation Technology Products AB, Sollentuna, Sweden).

An aluminum flow adapter cell was developed, installed in the loop and fitted with a pair of ultrasound transducers, 4 MHz (TN/TX, Imasonic, Bensancon, France). This flow adapter cell was used for measuring the flow profiles but also for acoustic measurements of the sound velocity (time of flight) and attenuation (peak-topeak voltage) of ultrasound. A digital oscilloscope (54624A, Agilent Technologies, Santa Clara, CA, USA) was an integral part of the data acquisition scheme.

Versatile MATLAB-based (The MathWorks Inc., Natick, MA, USA)

graphical user interface (GUI) software was developed to control all hardware devices, the data acquisition, signal processing and real-time monitoring of rheological properties. In-line flow profile measurements were made using a modified Doppler-based ultrasound velocity profiling instrument (UVP-Duo-MX, Met-Flow SA, Lausanne, Switzerland).

The modified UVP-Duo instrument was equipped with a firmware that allows access to the demodulated echo amplitude (DMEA). A new FFT based approach for estimation of the average Doppler shift frequency at each radial point was implemented and used in this work. The velocity profiles were then calculated by weighted averaging of the frequency spectrum.

The UVP-Duo instrument and the other hardware devices were connected to a master PC via Ethernet and DAQ card (National Instruments Sweden AB, Solna, Sweden). Communication with UVP hardware was implemented with an Active X library supplied by Met-Flow SA (Lausanne, Switzerland). More details are given in Wiklund et al.<sup>3, 4</sup>.

## Rheological in-line characterization

The velocity profile development was measured using the ultrasound Doppler velocity profiling technique. The corresponding pressure drop was measured in a section of pipe.and averaged over the time interval for each successive profile acquisition. In the present study, non-slip conditions, i.e. that the velocity is zero at the wall assumed for all investigated systems.

The velocity profile for a power-law fluid is obtained by integrating the equation above from the centre of the pipe to the pipe wall and is given by Eq. 1.

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

where r is a radial distance within the pipe calculated from the axis centre,  $\Delta P$  is the pressure drop over the distance L between the pressure sensors and R is the pipe radius. Here, K, is the power-law consistency index and, n, is the flow exponent.

Real-time least squares curve fitting of the velocity profiles and the pressure difference obtained to the integrated form of the power-law model was used to determine the parameters and the shear viscosity values during the liquid displacement processes.

## RESULTS

An example of an instantaneous measured velocity profile (red solid line) is shown with the corresponding power-law fit (black solid line) in Fig. 2 for stationary flow of Xanthan.



Figure 2. Instantaneous velocity profile (red solid line) with corresponding power-law fit (black solid line) for stationary flow of Xanthan.

Excellent agreement with conventional off-line rheometers was found for each system for a comparison over the shear rate range  $0.1-100 \text{ s}^{-1}$  and temperature matching the conditions in the experimental flow loop. A small discrepancy in the consistency index values for the industrial fluids can be explained by the strong dependence on the shear history of the samples. Details and obtained power-law parameters and shear viscosities are given in Wiklund et al.<sup>8</sup>.

The valve was turned to switch from Xanthan to sunflower oil after 30 s. An example of an instantaneous measured velocity profile (red solid line) is shown with the corresponding power-law fit (black solid line) in Fig. 3 for stationary flow of sunflower oil at the end of the displacement experiment. The change from the shearprofile thinning flat shape to the characteristic parabolic shape of а Newtonian fluid is clearly visible in Fig. 2 and 3.



Figure 3. Instantaneous velocity profile (red solid line) with corresponding power-law fit (black solid line) for stationary flow of sunflower oil.

Fig. 4 shows the corresponding development and variation in power-law parameters n, K, over time during the experiment.

Results showed that the shape of the velocity profiles remained the same but the magnitude changed about 2 s after the valve switch as the flow rate was affected by the sudden change in flow behavior and reduction in Reynolds number. The profile shape changed again after about 55s, which corresponds well to the estimated time required for the sunflower oil to reach the measurement position in the UVP flow adapter.



Figure 4. Corresponding variation in power-law parameters *n*, *K*, for the transition from Xanthan to sunflower oil.

The flow index *n* reached the expected value, n = 1, after about 75 s, as shown in Figure 4. In addition, Figure 4 shows that the consistency index, *K*, reached the expected value, K=0.06, at the same time, indicating a sharp transition zone from one immiscible fluid to the other and that a stable stationary flow was reached before data acquisition stopped. The UVP-PD method can thus be used to determine the time required for transition and length of the displacement zone.

The interface was found to be stable when the density of the displacing liquid was higher than the displaced one and when the fluids were immiscible and thus stabilized by surface tension effects. The converse displacement yield irregular profile shapes and much longer transition times.

The spatial distribution of phases was in some cases found to be invariably nonuniform due to e.g. differences in density. It was therefore essential to monitor the acoustic properties by continuously measuring the velocity of sound in the pipe cross-section.

#### CONCLUSIONS

Fast transient processes, such as liquid displacement of model and industrial fluids in pipes, can be monitored in-line, in realtime using the UVP-PD method developed. The results demonstrate the possibility to monitor the transition, from one fluid to another, both in terms of changes in the shape of the velocity profile and in rheological parameters.

# ACKNOWLEDGMENTS

This work is part of a PhD project financed by Vinnova, the Swedish Agency Innovation Systems. Tetra for Pak Processing System AB and Arla Foods. Gerard Gogniat and Olivier Mariette at Met-Flow SA, Switzerland are gratefully acknowledged for their technical support and scientific collaboration. Beat Birkhofer, Jeelani Shaik and Erich Windhab from ETH-Zurich, Switzerland, are gratefully acknowledged for scientific collaboration. Anna Oom and Ingrid Lindström are acknowledged experimental for their assistance. Luigi Messner (SIK workshop), Per Floberg and Lars-Göran Vinsmo are acknowledged for their technical assistance.

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