Optimization of the UVP+PD Rheometric Method for Flow Behavior Monitoring of Industrial Fluid Suspensions

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Abstract:
Ultrasonic Velocity Profiling (UVP) is a powerful technique for velocity profile measurements in research and engineering applications as it is the only available method that is cost-effective, relatively easy to implement and applicable to opaque fluid suspensions, which are frequently found in industry. UVP can also be combined with Pressure Drop (PD) measurements in order to obtain rheological parameters of non-Newtonian fluids by fitting theoretical rheological models to a single velocity profile measurement. The flow properties of complex fluids are almost exclusively obtained today using commercially available instruments, such as conventional rotational rheometers or tube (capillary) viscometers. Since these methods are time-consuming and unsuitable for real-time process monitoring, the UVP+PD methodology becomes a very attractive alternative for in-line flow behavior monitoring as well as quality control in industrial applications. However, the accuracy of the UVP+PD methodology is highly dependent on the shape and magnitude of the measured velocity profiles and there are still a few problems remaining with current instrumentation and methods in order to achieve the robustness and accuracy required in industrial applications. The main objective of this research work was to optimize an UVP+PD system by implementing new transducer technology and signal processing techniques for more accurate velocity profile measurements as well as rheological characterization of complex fluids under industrial/realistic conditions. The new methodology was evaluated in two different pipe diameters (22.5 and 52.8 mm) and tested with three different non-Newtonian fluids in order to obtain a wide range of rheological parameters. Results were also compared to conventional rotational rheometry and tube viscometry. It was found that rheological parameters obtained from accurate velocity data across the pipe radius, especially close to pipe walls where the velocity gradient is high, showed better agreement to conventional rheometry than when compared to results obtained using profiles measured with conventional UVP instrumentation and commercial software (Met-Flow SA Version 3.0). The UVP+PD method is now more robust and accurate. The main challenge remaining is to successfully implement a complete non-invasive system in industrial processes that is able to achieve real-time and accurate complex flow monitoring of non-Newtonian fluid suspensions.

Zusammenfassung:
Ultrasonic Velocity Profiling (UVP) ist eine schlagkräftige Methode zur Messung von Geschwindigkeitsprofilen in der Forschung und Technik. Es stellt zurzeit die einzige Methode dar, welche kostengünstig und einfach zu implementieren ist und Messungen in opaken liquiden Suspensionen ermöglicht, wie sie in industriellen Anwendungen häufig vorkommen. UVP kann mit Pressure Drop (PD) Messungen kombiniert werden und dadurch können mittels eines Fits eines einzigen Geschwindigkeitsprofils an theoretische rheologische Modelle die rheologischen Parameter der nicht-newtonschen Fluide ermittelt werden. Die Fließeigenschaften von komplexen Fluiden werden aktuell fast ausschließlich mit kommerziellen Instrumenten gemessen, z.B. mit dem konventionellen Rotationsrheometern oder Kapillarviskosimeter. Da diese Methoden zeitaufwändig sind und sich nicht für eine echt-zeit Prozesskontrolle eignen, stellt die Kombination der UVP+PD Methoden sowohl eine sehr attraktive Alternative für die In-Line Kontrolle der Fließeigenschaften dar als auch zur Qualitätskontrolle in industriellen Anwendungsbereichen. Die Genauigkeit der UVP+PD Methoden ist jedoch stark von der Form und der Größe der gemessenen Geschwindigkeitsprofile abhängig, so dass bei der jetzigen Geräteausstattung noch einige Probleme überwunden werden müssen um den von der Industrie geforderten Anforderungen an Robustheit und Genauigkeit entsprechen zu können. Das Hauptanliegen dieser Studie war die Optimierung eines UVP+PD Systems durch die Implementierung einer neuen Messwertgebertechnologie und Signalverarbeitungsmethodologie, um Geschwindigkeitsprofile mit einer höheren Genauigkeit zu messen und die rheologischen Eigenschaften von komplexen Fluiden unter industriellen/realistischen Rahmenbedingungen zu ermitteln. Diese neue Methodologie wurde in zwei unterschiedlichen Rohr-Durchmessern (22.5 & 52.8 mm) und an drei unterschiedlichen nicht-newtonschen Fluiden getestet und eine große Auswahl an rheologischen Parametern ermittelt. Die Resultate wurden mit konventionellen Messungen an Rotationsrheometern und Viskosimetern verglichen. Die durch die neue Methodologie einer genaueren Geschwindigkeitsprofilmessung erhaltenen rheologischen Para-

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meter zeigen eine größere Übereinstimmung mit den konventionellen Messungen als die Messungen mit konventionellen UVP Messsystemen und der der kommerziellen Software (Met-Flow SA Version 3.0). Die UVP+PD Methode ist nun robuster und genauer. Die größte zukünftige Herausforderung stellt nun die erfolgreiche Implementierung eines kompletten, nicht-invasiven Systems in einen industriellen Prozess dar, um eine Echtzeit Überwachung der Fließeigenschaften von nicht-newtonschen Fluiden von hoher Genauigkeit zu erhalten.

Résumé:
La vélocimétrie (Doppler) ultrasonore (VDU) est une technique puissante de mesure de profil de vitesse pour les applications en recherche et d'ingénierie. C´est en effet la seule méthode disponible qui est rentable, relativement facile à mettre en œuvre et applicable à des suspensions de fluides opaques, communs dans l'industrie. VDU peut également être combinée avec des mesures de perte de charge (PC) afin d´obtenir des paramètres rhéologiques de fluides non-newtoniens en faisant correspondre les prédictions de modèles rhéologiques théoriques à une mesure unique de profil de vitesse. Les propriétés d'écoulement des fluides complexes sont, aujourd'hui, presque exclusivement obtenues en utilisant des instruments disponibles dans le commerce, tels que des rhéomètres rotatifs ou des viscosimètres capillaires. Puisque ces méthodes prennent du temps et ne sont pas applicables au contrôle de procédés en temps réel, la méthode combinant VDU et PC offre une alternative attrayante pour le contrôle en ligne du comportement d'écoulement ainsi que le contrôle de la qualité dans les applications industrielles. Toutefois, la précision de la méthode combinant PVU et PC dépend fortement de la forme et de l'ampleur des profils de vitesse mesurés et il y a encore quelques problèmes avec l´instrumentation et les méthodes pour obtenir la robustesse et la précision qui est requise dans les applications industrielles. L'objectif principal de ce travail de recherche est d'optimiser un système combinant VDU et PC en mettant en œuvre une nouvelle technologie des transducteurs et des techniques de traitement du signal pour obtenir des mesures de profil de vitesse plus précises ainsi qu´une caractérisation rhéologique des fluides complexes dans des conditions industrielles / réalistes. La nouvelle méthodologie a été évaluée dans deux tubes de diamètres différents (22,5 et 52,8 mm) et testée avec trois différents fluides non-newtoniens en vue de couvrir un large éventail de paramètres rhéologiques. Les résultats ont été également comparés avec ceux obtenus par rhéométrie rotative classique et par viscosimétrie capillaire. Il a été constaté que les paramètres rhéologiques obtenus à partir des données précises de vitesse le long du rayon du tube, en particulier près des parois où le gradient de vitesse est élevé, sont en meilleur accord avec la rhéométrie classique que ceux déterminés en utilisant des profils mesurés avec une instrumentation PVU classique et des logiciels commerciaux (Met-Flow SA version 3.0). La méthode PVU + PC est maintenant plus robuste et plus précise. Le défi principal restant est de mener à bien l´implémentation dans les procédés industriels d´un système complet et non-invasif étant capable de réaliser en temps réel un contrôle précis du flux de fluides non-newtoniens

Key words: Ultrasonic Velocity Profiling, UVP-PD methodology, rheology, non-Newtonian, tube viscometry

1 INTRODUCTION

Ultrasonic Velocity Profiling (UVP) is originally a medical technique for measuring an instantaneous velocity profile in liquid flow along the pulsed ultrasonic beam axis. The instantaneous velocity profile is obtained by detecting the relative time lags between pulse emissions echoed by particles contained in the fluid as a function of time. The UVP technique for determining a one-dimensional velocity profile in fluid flow was introduced for general fluids by Takeda [1, 2]. The UVP technique and its applications in academia and industry is now well described in several scientific publications, see [3–6]. The UVP technique can be combined with Pressure Difference (PD) measurements in order to obtain in-line rheological parameters of opaque fluids with suspended particles and is usually referred to as the UVP+PD methodology [5, 7]. It is based on the traditional tube viscometry concept where the shear rate is obtained from the measurements of the volumetric flow rate and the shear stress at the wall is obtained from simultaneous measurement of the pressure difference over a fixed distance. The UVP+PD method and similar methods for determining the rheological parameters of a suspension or emulsion are already known and is described in several publications [5, 7–17].

All systems presented in the literature up to around 2008 were more or less based on off-the-shelf transducers and electronics and could thus only be used for simple flow characterization

with limited accuracy and without meeting industrial requirements. Extensive work was done in recent years by several research groups in order to improve the performance, the user friendliness and the accuracy and robustness of the UVP+PD system and methodology. For example, new software with a graphical user interface (GUI) was developed that provides a complete tool for data acquisition from all hardware devices (pressure sensors, velocity of sound measurements and temperature). The software was based on Matlab® and the UVP-DUO Active X libraries driver for communication with the electronics [5, 7, 11, 12, 18]. An extended and complete commercial UVP+PD software, RheoFlow™ was later developed, see for example Wiklund and Stading [14], Wiklund *et al.* [19] and Wassel *et al.* [17], which is capable of rapid data processing and can serve as a basis for in-line real-time process monitoring of rheological properties and also accurate real-time characterization of a wide range of different fluids under true process conditions.

Apart from the user interface and signal processing software, current ultrasonic transducer technology also adds more limitations to the UVP+PD system. Two possible transducer installation methods exist, non-invasive (i.e. through-the-wall measurements) or flush mounted. Clamp-on Doppler and transit-time flow meters are commercially available with accuracies in the 1–2% range (application dependent). However, most of these instruments cannot measure instantaneous radial velocity profiles or accurate velocity gradients near the wall due to the sensor design and flow adapter configurations. Consequently, the most widely used installation method is flow adapters that enable direct contact with the transducer surface and fluid medium (see Figure 2), which ensures minimal wave refraction and ultrasound attenuation [5 ,7]. Current transducer designs exhibit long near-field distances, e.g. 30–17 mm for 2–4 MHz standard transducers [20]. Accurate velocity measurements are not possible within the near-field distance due to the highly irregular pressure field extending to the focal point and thus transducers are installed with a distance away from the pipe wall interface. Determining the actual wall interface (when measuring through material layers) or liquid-wall interface (when measuring with direct contact to the test fluid) is of great importance for rheological measurements. The determination of the interface is extremely complicated, especially when measuring velocity profiles with limited spatial resolution or when attenuation distorts the quality of near wall velocity data. It has been shown that by changing the wall position by less than 0.37 mm (or one channel) the rheological parameters determined using the UVP+PD method vary significantly [5, 7]. This is since the fluids under investigation are subjected to, and most influenced by, the strongest shear in the near-wall region with highest velocity gradients as described above. The problem of uncertain wall positions has forced users to obtain rheological data of the test fluid using other methods such as off-line rotational rheometry in order to adjust wall interface positions that yield the correct fluid properties [13, 21]. This defies the purpose of the UVP+PD methodology for in-line rheometry, as one would like to develop a complete UVP+PD based measuring system and methodology which can measure rheological properties without any *a priori* knowledge of the fluid characteristics. Furthermore, using standard transducer and installation techniques (as shown in Figure 2) prevents any measurements in fluids that attenuate ultrasonic energy, which is frequently found in industrial fluid suspensions [22].

The main objective of this research work was to optimize and describe an UVP+PD system suitable for real-time rheological characterization of industrial fluids directly in-line while under process conditions. Results were compared with that obtained using previous UVP+PD equipment and software as well as using conventional rotary rheometry and tube viscometry.

2 THEORY

This section briefly describes non-Newtonian flow and the associated rheological parameters that were used to characterize the fluid suspensions in this work. The equation for the Herschel-Bulkley model is as follows:

$$\tau = \tau_y + K(\dot{\gamma})^n \tag{1}$$

where K, n and τ_y are three curve-fitting parameters [23]. Equation 1 can be integrated to give the velocity v profile across the pipe radius:

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

where R_{plug} is the plug radius and is related to the fluid yield stress according to:

$$R_{plug} = \frac{2L\tau_y}{\Delta P} \quad (3)$$

The Herschel-Bulkley model can easily be modified to describe the power-law and Bingham plastic models [23]. The identification of the transition between laminar and turbulent flow is of great importance because the fluid flow behavior changes fundamentally at the transition zone. Slatter and Lazarus [24] formulated a Reynolds number Re_2 for non-Newtonian pipe flow:

$$Re_2 = \frac{8\rho V^2}{\tau_y + K\left(\frac{8V}{D}\right)^n} \quad (4)$$

where ρ is the density, V the bulk velocity, and D the pipe inner diameter. Note that in order to calculate rheological parameters all velocity profiles were measured in laminar flow. Equation 4 was used as an indication of the flow regimes in which tests were conducted in this work.

3 MATERIALS AND EXPERIMENTAL METHODS

3.1 MATERIALS

Carboxy Methyl Cellulose (CMC) was tested and is generally regarded as an ideal non-Newtonian power-law fluid for experimental work, especially when ultrasonic tests are concerned as CMC offers excellent wave scattering/reflection and minimal attenuation [7]. The CMC (Protea Chemicals, Bryanston, South Africa, http://www.proteachemicals.co.za) solutions used were 6.15 and 6.8% w/w. Bentonite powder (Protea Chemicals) was mixed with water to obtain different concentrations of bentonite:water suspensions. The concentration tested was 8% w/w. Dry kaolin powder (Protea Chemicals) was used to prepare a kaolin: water suspension of 13% v/v. Kaolin suspensions attenuate the ultrasonic energy significantly [7] and was thus used to test the limitations of profile measurements using different ultrasonic transducers. These fluids were selected in order to yield a wide range of rheological properties for characterization of complex flow behavior using different techniques.

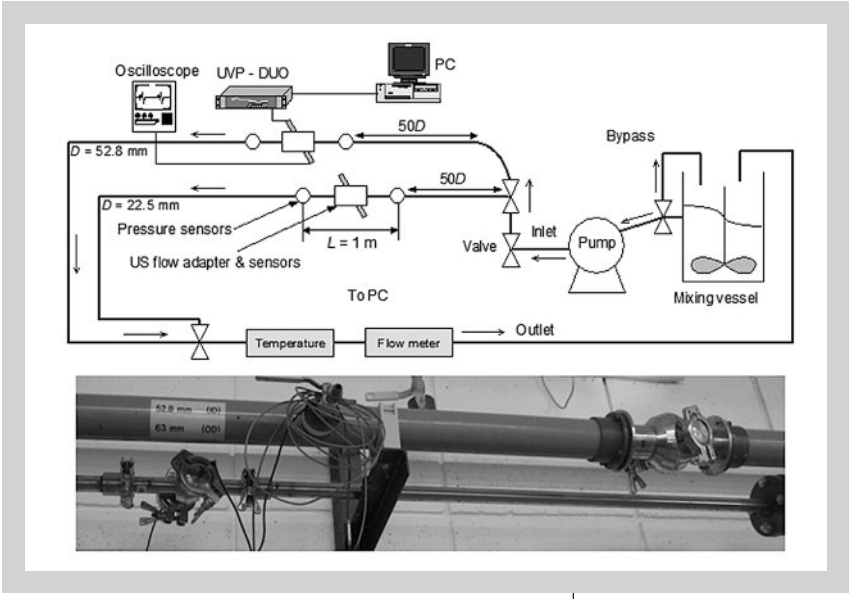


Figure 1: Schematic illustration of the UVP flow loop and flow adapters installed on the pipe rig.

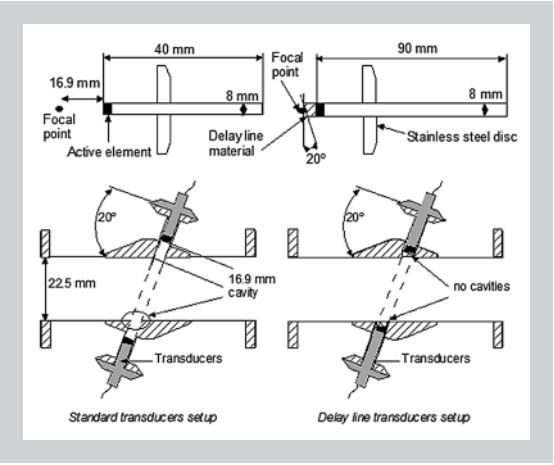
3.2 UVP+PD FLOW LOOP AND TUBE VISCOMETER

The pipe rig consisted of three Polyvinyl chloride (PVC) pipes with inner diameters of 52.8, 43, and 22.5 mm and was used for tube viscometry tests. The wall shear stress data was obtained from pressure drop measurements ($D\Delta P/4L$) and the nominal wall shear rate from flow rate measurements ($8V/D$) which was corrected for by the Rabinowitch-Mooney factor. Procedure and method for obtaining accurate in-line experimental data as well as post data analysis is discussed in detail by Chhabra and Richardson [23, 25]. The pipe rig was fitted with a thermocouple (accuracy ± 1°C) and 50 mm flow meter (Krohne IFC 010D – DN40) with an accuracy (for water) of 0.5% of the measured value for V ≤ 0.4 m/s and 0.002 m/s for V > 0.4 m/s. Pressure measurements were conducted using differential pressure transducers (Fuji Electric) with maximum range of 130 kPa and an accuracy of 0.25%. A similar setup can be found in Fester *et al.* [26]. UVP flow adapters were installed in-line in the 52.8 and 22.5 mm pipes for rheological characterization of the test fluids using the UVP+PD methodology. Figure 1 shows a schematic diagram of the UVP flow loops and the two flow adapters installed in the pipe rig.

3.3 UVP+PD EXPERIMENTAL SETUP

In this work a commercial UVP instrument (UVP-DUO-MX, Met-Flow SA, Lausanne, Switzerland)

Figure 2:
Standard and delay line transducers and installation setup [22].

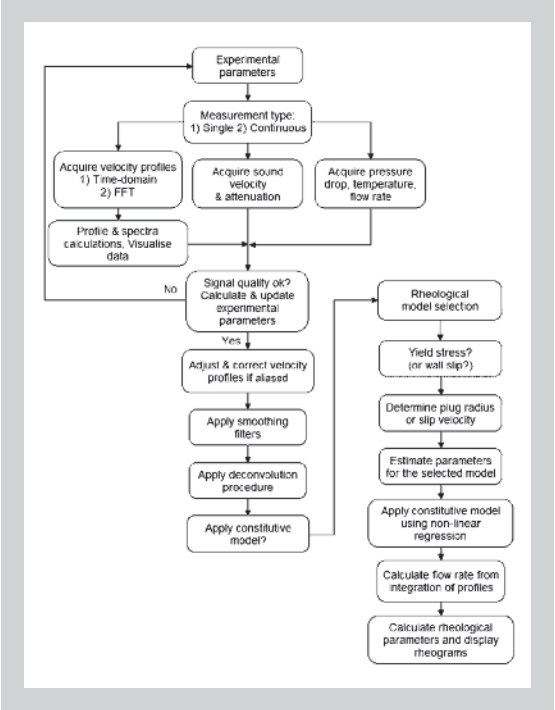


was used for velocity profile measurements. A more detailed description, as well as technical information about the UVP instrument, can be found in Met-Flow SA [20]. A new transducer, which incorporates a delay line, was used for velocity profile measurements. The delay line is a material optimized for beam forming that contains the near-field distance. This delay line is fixed ahead of the transducer and is in flush with the pipe wall, thus making it possible to have the focal point of the ultrasonic beam at the wall interface, see Wiklund [12] and Kotzé and Haldenwang [22]. Transducers with a central basic frequency of 4 MHz were chosen in order to obtain good compromise between spatial resolution, which is due to their short wavelength, and penetration depth (less attenuation). The 4 MHz sensors have a fixed active beam diameter of 5 mm and were used for velocity profile as well as sound velocity measurements. Doppler, Immersion type 4 MHz, 5 mm active element standard ultrasonic transducers (TN and TX-line, Imasonic, Bensancon, France) were used for comparison with results obtained using the delay line transducers. Technical information regarding the standard transducers can be found in Met-Flow SA [20]. A special flow adapter cell made from stainless steel was designed for simultaneous in-line measurements of velocity profiles and acoustic properties for straight pipe flow. Ultrasonic Transducers (TDX) were installed at 20° with respect to the lateral direction (see Figure 2) and in direct contact with the fluid to avoid attenuation and reflection of the pressure wave. To avoid measurements where the ultrasonic pressure wave is irregular, the standard transducers were also pulled back creating a cavity equal to the transducer near field distance (~ 17 mm) between the transducer surface and actual pipe wall interface. This transducer installation has previously been described in Wiklund *et al.* [5] and the same setup was used in Kotzé *et al.* [7]. Figure 2 shows a schematic diagram of the flow adapters with standard and delay line transducers.

An Agilent 100 MHz Digital Oscilloscope (Model 54622A) was used as an integral part of the set-up for velocity of sound measurements and the procedure has been described in previous work [5]. The velocity of sound parameter was used to calculate the correct magnitude of the velocities measured as well as the spatial positions along the measurement axis (ultrasonic beam). The velocity of sound was monitored continuously as it changes with temperature and consequently influences the magnitude of measured velocities.

3.4 UVP+PD PROCESSING STEPS

Regardless of the actual setup (UVP and ultrasound equipment), the optimized methodology requires several processing steps. A flowchart of the data acquisition and processing steps involved is given in Figure 3. The whole process is automated and can be performed in real-time. Experimental parameters (echo data, sound velocity, pressure, temperature, flow rate, pulse waveforms, attenuation) are recorded using a digital data acquisition device with interface to a PC. Direct access to Demodulated Echo Amplitude (DMEA) data enables the user to implement custom velocity estimation algorithms, such as time and frequency domain based algorithms [5]. Spectral analysis proved to be valuable as it provided quality information such as signal amplitude and signal-to-noise ratio, but also simplified identification of signal artifacts and causes of velocity errors. Errors caused by aliasing can also be corrected for by applying error correction software to the measured data. After data acquisition, the quality of the measured data can be enhanced by applying various smoothing filters (such as Singular Value Decomposition, Finite Impulse Response, Infinite Impulse Response and Moving Average filters), which can be selected by the user using a GUI. Commercial UVP instruments usually employ one simple type of low pass filter, which is typically incorporated into hardware or the instrument's Digital Signal Processor (DSP). Depending on the application, this could result in noisy and bad quality data, which can result in significant errors in profile measurements and ultimately the rheological parameters determined using the UVP+PD methodology. After velocity profiles are calculated and visualized by the software a deconvolution procedure is applied in order to correct



near wall velocity data and gradients for accurate calculation of flow rates (by integration) as well as rheological parameters. The deconvolution procedure is discussed in detail by Kotzé and Haldenwang [22]. Once the accuracy and quality of the experimental parameters (pressure, echo data, velocity profiles) have been established to be at an acceptable level, the rheological parameters, volumetric flow rate and other parameters such as attenuation properties and solids concentration are calculated. The user has access to different non-model and model fitting techniques and rheological models (such as the power-law, Bingham, Herschel-Bulkley, Sisko, Casson, Cross, Ellis, Carreau or similar models) in the commercial UVP+PD software, RheoFlow™ by Wiklund and Stading [14]. The new signal processing techniques in combination with delay line transducers also ensured fixed wall interface positions which are crucial for accurate and independent rheological characterization of non-Newtonian fluids [22].

3.5 ROTARY VISCOMETER

The conventional rheometer used at the Material Science and Technology (MST) group was a Paar Physica MCR300 (Anton Paar, Randburg, South Africa, www.advancedlab.co.za) instrument which was equipped with an air bearing. The configuration that was used to test the mineral suspensions was the cup and bob geometry. Flow curves were created for CMC, bentonite and kaolin suspensions and the results were compared with in-line tube viscometry as well as with that obtained using the UVP+PD method.

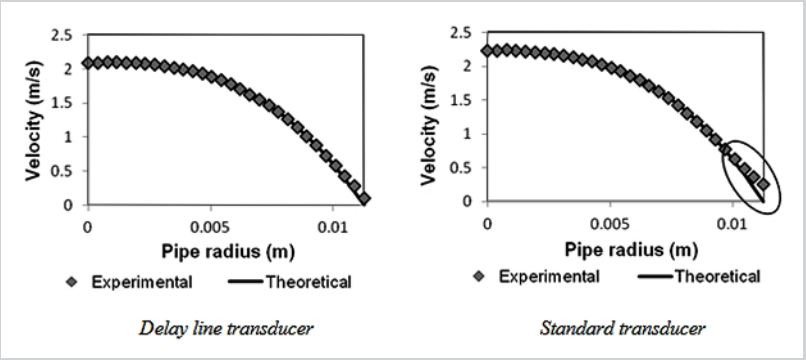


Figure 3 (left): UVP+PD data processing structure (adapted from Wiklund et al. [5]).

Figure 4: Experimental and fitted theoretical velocity profile for delay line and standard transducer (22.5 mm pipe, CMC 6.8 % w/w).

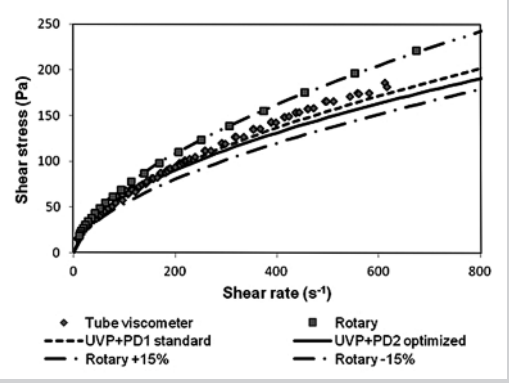
4 RESULTS AND DISCUSSION

This section compares results obtained using the optimized (delay line transducer and new signal processing techniques) as well as standard (old transducers and no new signal processing) UVP+PD methodology. Results are compared to that obtained from conventional rheometric methods, in-line tube viscometry and off-line rotational rheometry. Two UVP+PD flow loops of different diameters (22.5 and 52.8 mm) were constructed for comparison and to test system limitations. The tests were conducted with different concentrations of bentonite and kaolin suspensions as well as CMC solutions.

4.1 UVP+PD MEASUREMENTS IN 22.5 MM PIPE

4.1.1 Comparison of different rheometric methods for CMC 6.8 % w/w

Figure 4 compares the experimental data measured using the delay line and standard transducer with a theoretical velocity profile (Equation 2) determined from a least-squares fit. The measurements were conducted in CMC 6.8 % w/w at a flow rate of Q = 0.55 l/s (Re_2 = 108). The wall positions of the power-law profiles measured using standard transducers were calculated by using the maximum velocity as the centre position and subtracting the pipe radius. This method was used for both pipe diameters (22.5 and 52.8 mm). The wall position calibrated (from maximum centre velocity) using the power-law fluid CMC was assumed correct and kept constant for velocity profile measurements in the bentonite and kaolin suspensions. Fixed wall positions (length of delay line material, Figure 2) were used for all velocity profiles measured using delay line transducers. By fitting a theoretical velocity profile onto experimental data the parameters K, n, and τ_y are solved. A flow curve is constructed and compared to rheological data obtained from tube and rotary viscometry. In this work UVP+PD1 refers to the method where a standard transducer was used and UVP+PD2 to the optimised system and methodology, i.e. delay line transducers and advanced signal processing steps.



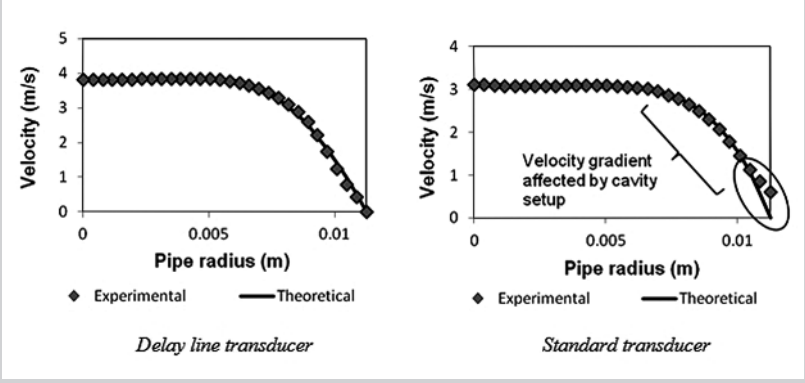
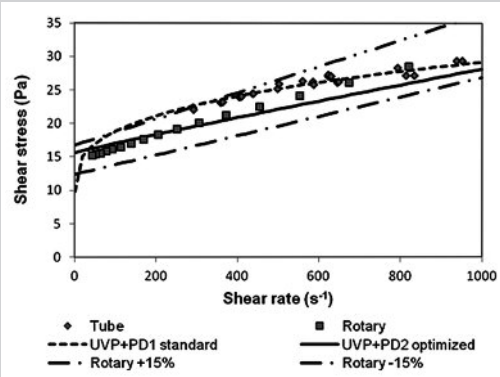


Figure 5 (left above): Rheogram for CMC 6.8% w/w (22.5 mm pipe).

Figure 6 (right above): Experimental and fitted theoretical velocity profile for delay line and standard transducers (22.5 mm pipe, bentonite 8 % w/w).

Figure 7 (below): Rheogram for bentonite 8% w/w (22.5 mm pipe)



Rheological results are schematically illustrated in Figure 5 and the parameters determined from a power-law fit (Equation 2) are shown in Table 1. It can be observed that non-zero velocities were measured (shown by circle in Figure 6) using the standard transducer due to the cavity setup. However, this did not affect the outcome of the final result, as shown by the striped line in Figure 7. Good agreement of ± 15 % was found between all the rheometric methods. According to Tab. 1, the goodness-of-fit (R^2) was very good for all the power-law curve fittings, except for the UVP+PD1 results, where the cavity affected the fitting of the velocity profile.

4.1.2 Comparison of different rheometric methods for bentonite 8 % w/w

An experimental (obtained using standard and delay line transducers) and theoretical velocity profile for bentonite 8 % w/w is compared in Figure 6. Tests were conducted at a flow rate of Q = 1 l/s (Re_2 = 1764). Bentonite suspensions showed little attenuation and absorption of ultrasonic energy and thus the acoustic energy could penetrate across the whole pipe diameter. Generally a good echo signal was obtained for all concentrations of bentonite. The cavity in front of the standard ultrasonic transducer influences the quality of the measured velocity profile close to the wall interface (shown by circle in Figure 6). Also, the gradient of the velocity profile (Figure 6) was affected due to the combination of the plug flow present for bentonite suspensions (characterized by Bingham model) and large cavity to pipe diameter ratio (8 mm/22.5 mm). As a result an erroneous plug radius was obtained, which consequently affects the profile fitting and determination of rheological parameters. Non-zero velocities at the wall interface are forced to zero in order to optimise the fitting procedure for calculating rheological parameters. This unfortunately does not improve the overall accuracy of the fit. Furthermore, since there are non-zero velocities at the pipe wall it becomes extremely difficult to estimate wall positions. A significant variation in rheological parameters is observed when wall positions are changed by only one channel distance (less than 0.35 mm) [5, 7]. Calculation of wall positions from plug flow profiles becomes even more complicated due to maximum velocities present, beyond the pipe radius. Even when a correct wall position could be obtained for example by visual inspection or post data analysis, the theoretical fitting onto the experimental velocity profile is not straight forward and thus far users still need to adjust boundary conditions and initial estimates in order to obtain the correct rheological parame-

Table 1 (left): Rheological parameters measured in 22.5 mm pipe for CMC 6.8 % w/w.

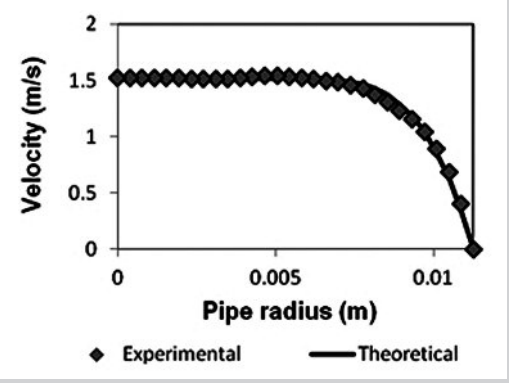
Rheometric method	K (Pa.s)	n -	τ_y (Pa)	R^2
UVP+PD1	4.82	0.56	0	0.992
UVP+PD2	5.22	0.54	0	0.998
Tube viscometry	4.44	0.58	0	0.997
Rotary viscometry	4.41	0.61	0	0.999

Table 2 (middle): Rheological parameters measured in 22.5 mm pipe for bentonite 8 % w/w.

Rheometric method	K (Pa.s)	n -	τ_y (Pa)	R^2
UVP+PD1	1.95	0.33	9.84	0.986
UVP+PD2	0.019	0.94	15.58	0.997
Tube viscometry	0.0097	1	20.1	0.947
Rotary viscometry	0.017	1	14.59	0.999

Table 3 (right): Rheological parameters measured in 22.5 mm pipe for kaolin 13 % v/v.

Rheometric method	K (Pa.s)	n -	τ_y (Pa)	R^2
UVP+PD2	0.48	0.37	5.7	0.966
Tube viscometry	0.41	0.46	4.1	0.995
Rotary viscometry	0.61	0.42	4.5	0.995



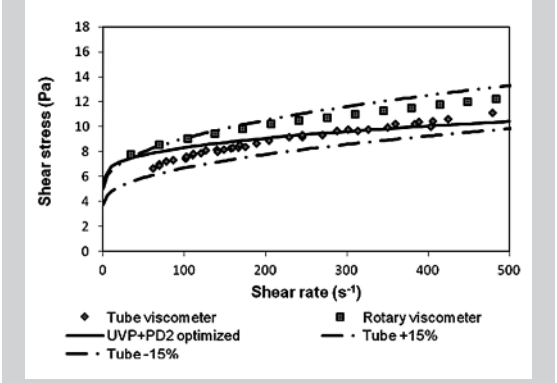


Figure 8 (left): Experimental and fitted theoretical velocity profile for delay line transducer (22.5 mm pipe, kaolin 13 % v/v)

Figure 9: Rheogram for kaolin 13 % v/v (22.5 mm pipe)

ters. Other scientists have tried different approaches to the model fitting procedure such as using polynomials of different orders, irrational power equations, splines or using different rheological models, see e.g. Pfund *et al.* [10], Wunderlich and Brunn [27] as well as Ouriev [28]. The problem of requiring knowledge of boundary conditions and initial estimates and/or fitting of different models forces the user to have knowledge of the fluid characteristics beforehand, which as explained earlier, do not make this a viable independent rheometric measurement method.

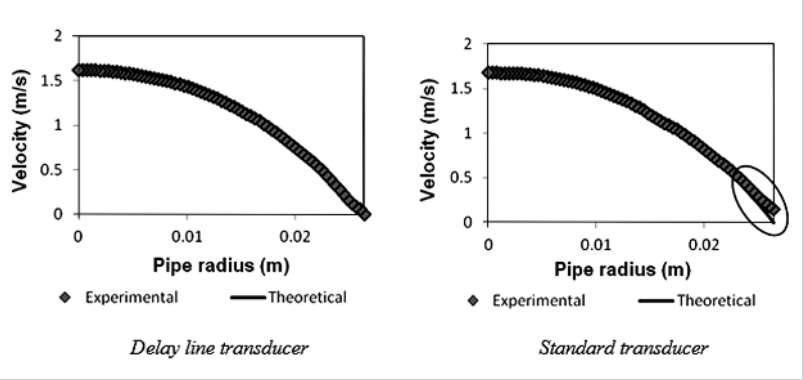
From Figure 7 it can be observed that results obtained from tube viscometry and UVP+PD2 are in good agreement (within 15 %) with each other across the relevant shear rate region (300 to 1000 s^{-1}). However, the flow curve obtained from UVP+PD1 suggests a yield pseudoplastic fluid with a low yield stress. When compared to tube and viscometer data this is clearly not the case. The flow curve obtained from the rotary rheometer showed lower apparent viscosities. This could have been due to the time dependant properties of the bentonite suspensions. Although samples were pre-sheared before measuring flow curves, this did not always yield good comparative results. This also illustrates the importance of determining rheological properties in-line during process conditions, as the rheological properties of fluid samples may often change with time.

An important observation is the difference in yield stress measurements using UVP+PD1 and UVP+PD2 (see Table 2). The yield stress is calculated from direct measurements of the plug radius and pressure drop. Due to inaccurate velocity gradients at the pipe wall the plug radius is affected and thus the yield stress measurement. Also, when observing tube viscometry results it can be seen that data were not available at the lower shear rates. This was due to limitations in the pressure drop measurements and in this case the UVP+PD2 methodology was able to characterise the fluid accurately across the relevant shear rate range.

4.1.3 Comparison of different rheometric methods for kaolin 13 % w/w

Figure 8 illustrates experimental and theoretical profiles measured using the delay line transducer for kaolin 13 % v/v (Q = 0.52 l/s, Re_2 = 1521). It was not possible to measure velocity profiles using the standard transducer setup as the kaolin suspension filled the cavity and absorbed all of the acoustic energy [7]. The theoretical fit showed good correlation with the experimental data, especially close to the wall, where the velocity gradient is high.

Figure 9 shows the rheogram obtained from tube (in-line) and rotary (offline) viscometry, as well as using the UVP+PD2 methodology. Tab. 3 shows the rheological parameters determined from a Herschel-Bulkley model fit. There is good agreement (less than 15%) between the tube viscometer and UVP+PD2 results. Here the same limitations were found when using the tube viscometer as no apparent viscosities could be measured across the lower shear rate range (< 100 s^{-1}). The rotary viscometer could measure across lower shear rates and therefore showed more curvature (for shear rates lower than 100 s^{-1}) than when compared to the UVP+PD2 method. In this case the user could assume that the UVP+PD2 method is more representative of the actual system, since the rheology was obtained in-line under true test conditions. From the fitting procedure (using the Herschel-Bulkley model) it can be seen that the yield stress measurements shown in Table 3 were in good agreement. It is therefore critical that the plug radius is measured correctly by achieving accurate velocity data at the wall interface in order for the in-line UVP+PD2 method to work. In this case the optimized UVP+PD2 method was able to produce information of the flow properties of a fluid suspension which significantly attenuates and absorbs ultrasonic energy. These types of complex fluids are frequently found in industrial applications and thus the new setup using delay line transducers proved to be very encouraging for future implementation in a wide range of fluid engineering applications.



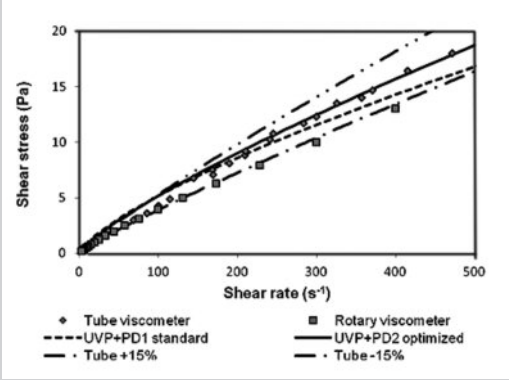


Figure 10 (left): Experimental and fitted theoretical velocity profile for delay line and standard transducers (52.8 mm pipe, CMC 6.15 % w/w).

Figure 11: Rheogram for CMC 6.15 % w/w (52.8 mm pipe)

Table 4 (left below): Rheological parameters measured in 52.8 mm pipe for CMC 6.15 % w/w

Table 5 (right below): Rheological parameters measured in 52.8 mm pipe for bentonite 8 % w/w

4.2 UVP+PD MEASUREMENTS IN 52.8 MM PIPE

4.2.1 Comparison of different rheometric methods for CMC 6.15 % w/w

Figure 10 compares the experimental data measured using the delay line and standard transducer with a theoretical velocity profile (Equation 2) determined from a least-squares fit. The measurements were conducted in CMC 6.15 % w/w at a flow rate of Q = 2.02 l/s (Re_2 = 1440). Similar results as for the UVP+PD measurements in the 22.5 mm pipe were found for the CMC solution in the larger diameter pipe (52.8 mm). The influence of the cavity (shown by circle in Figure 10) did not affect the accuracy of rheological parameters, which are illustrated in Figure 11 and shown in Table 4. All the results are within 15 % with the tube viscometer across the entire shear rate range.

4.2.2 Comparison of different rheometric methods for bentonite 8 % w/w

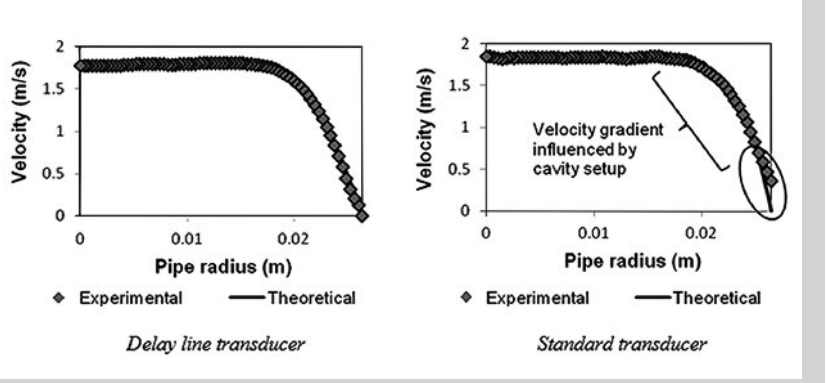
As explained before, the cavity influences the quality of the measured velocity profile and thus it becomes difficult to fit theoretical velocity profiles to experimental data without error. An experimental (delay line and standard transducer) and theoretical velocity profile for bentonite 8% w/w (similar fluid used for 22.5 mm pipe tests) is compared in Figure 12. Tests were conducted at a flow rate of Q = 3.25 l/s (Re_2 = 1001).

Figure 13 and Table 5 summarizes the rheological results and parameters obtained from conventional rheometric methods and using the two UVP+PD methodologies. Again good agreement was found when comparing the UVP+PD2 method (delay line and deconvolution combination). The results obtained using the standard transducer configuration (UVP+PD1) show a yield-pseudoplastic model with a low yield stress, as previously found for results obtained in the 22.5 mm diameter pipe (bentonite suspension). It should be, however, mentioned again here that accurate rheological parameters could be obtained from the UVP+PD1 method by using different wall positions, that is by shifting the profile so that a larger plug radius can be obtained. Even a margin of 0.5 mm results in similar results as found with the UVP+PD2 method. In this case the user need to have knowledge of the rheology of the fluid beforehand in order to see what wall position is correct (initially the wall positions for standard transducers were calculated from profiles measured in CMC solutions and kept fixed (Section 4.1). By using the delay line transducers no knowledge of the fluid properties are required *a priori* as fixed wall interface positions are used, no matter what fluid is under investigation.

Good correlation was found between the yield stress measurements obtained from the UVP+PD2 method and off-line rheometry (see Table 5). The yield stress obtained from the Bingham model fit to the tube viscometry data was higher and since no data could be obtained in the low shear rate region (< 300 s^{-1}), the result obtained from the UVP+PD2 method could be assumed more correct. Unfortunately it was not possible to measure profiles in kaolin suspensions in the 52.8 mm pipe as the delay line transducers could not penetrate across the pipe radius (R = 26.4 mm), which is the minimum penetration depth required for in-line rheological characterization (only half of the experimental velocity profile is needed). A transducer with a fixed delay line material which would absorb less acoustic energy could solve the previously mentioned problems. This is currently under investigation.

Rheometric method	K (Pa.s)	n -	τ_y (Pa)	R^2
UVP+PD1	0.18	0.73	0	0.994
UVP+PD2	0.13	0.81	0	0.999
Tube viscometry	0.077	0.89	0	0.996
Rotary viscometry	0.086	0.83	0	0.999

Rheometric method	K (Pa.s)	n -	τ_y (Pa)	R^2
UVP+PD1	3.2	0.216	8.73	0.975
UVP+PD2	0.026	0.97	13.75	0.998
Tube viscometry	0.0097	1	20.1	0.947
Rotary viscometry	0.017	1	14.59	0.999



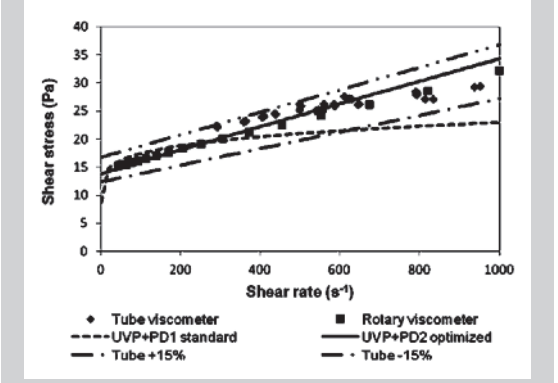


Figure 12 (left): Experimental and fitted theoretical velocity profile for delay line and standard transducers (52.8 mm pipe, bentonite 8 % w/w)

Figure 13: Rheogram for bentonite 8 % w/w (52.8 mm pipe)

5 CONCLUSIONS AND RECOMMENDATIONS

This article presents an optimized UVP+PD methodology for accurate and independent characterization of complex fluids in industrial applications. This was achieved by using delay line transducers in combination with optimized signal processing techniques capable of several velocity estimation methods and a deconvolution procedure, which lead to more accurate velocity gradients in the near wall region. Rheological parameters obtained from more accurate profile gradients close to pipe walls showed better agreement with conventional rheometric methods (within 15 %).

In comparison, a conventional set-up with flush-mounted standard transducers and small cavities in front of the transducer prevented measurements in attenuating fluids as the fluid filled up the entire cavity and absorbed the ultrasonic energy, as found with the kaolin suspensions in the 22.5 mm pipe. The delay line transducers eliminated this problem and showed that this methodology (combination of new transducer technology and advanced signal processing techniques) is essential for accurate monitoring of non-Newtonian fluid behavior as well as in-line rheology in industrial applications, where complex fluids with attenuating properties are encountered frequently.

The presented results show that the UVP+PD in-line rheometric method in combination with an invasive set-up with delay line transducers is now more versatile (applicable in wide range of pipe diameters and fluids), robust (measurements in attenuating fluids) and accurate (no knowledge of rheology initially is required). However, there are still a few limitations remaining. The delay line material used here absorbs ultrasonic energy and prevents measurements in larger pipe diameters in attenuating fluids. Although the signal processing techniques ensured accurate velocity data, the current setup produced rheograms and velocity profiles after about 30 – 60 seconds of calculation time. The data processing scheme and software (see Figure 3) need to be improved in order to obtain results in real-time for monitoring and control of dynamic processes. Limitations with the current delay line technology such as, temperature, pressure, high attenuation and wear resistance must also be addressed. New transducer development is required so that maximum energy transfer into the fluid medium is possible which will increase the penetration depth and applicability in industrial applications where large diameter pipelines are used.

The main remaining challenge is to find an optimal sensor setup for industrial applications. A non-invasive clamp-on set-up is an attractive alternative solution as industrial processes constantly run day and night and it is often not possible to stop these for installation and calibration of UVP equipment. Another significant advantage is that these transducer setups would not be directly exposed to high pressures, temperatures and harsh conditions found in industry. However, this setup could lead to new installation (mounting of sensors and pipe vibrations) and calibration (acoustic coupling) problems. Clamp-on systems have existed for more than 20 years and so far it has only been successful for accurate transit-time (bulk flow rate) and not Doppler measurements. The reasons for this are due to ultrasonic beam refraction, loss of energy, unknown sample volume shapes and wall interface positions after material layers that complicate measurements and results in inaccurate flow profile measurements [22]. New optimized sensor solutions are needed in order to completely implement the UVP+PD method in a dynamic and robust industrial environment.

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