

IN-LINE ULTRASOUND BASED RHEOMETRY OF INDUSTRIAL AND MODEL SUSPENSIONS FLOWING THROUGH PIPES

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ABSTRACT

A pulsed ultrasound (U) Doppler based technique is experimentally evaluated for in-line rheometry of viscoelastic surfactant solutions (shampoo) and non-Newtonian aqueous cellulose fibre suspensions flowing in a pipe over a wide range of volume throughputs and concentrations. The method involves the simultaneous measurement of radial velocity profile (VP) determined by the time delay and frequency of ultrasound reflected by particles, and pressure drop (PD) in a pipe section. The experiments are carried out in a flow loop fitted with a flow adapter having optimum acoustic properties, a transducer, which emits ultrasound at a frequency of 4 MHz and receives the echo signal, and a Met-Flow UVP-Monitor. For each volume throughput, a non-linear regression analysis of the velocity profile measured by the in-line UVP-PD rheometer is carried out using the integrated form of power-law rheological model to obtain the exponent and consistency index for both surfactant solutions and cellulose suspensions. In addition, Herschel-Bulkley model is used to obtain the yield stress for cellulose suspensions. The model parameters and the shear rate dependent viscosities for the shear thinning surfactant solutions determined using the in-line UVP-PD technique agree well with those obtained using the conventional commercial rotational off-line rheometers (Bohlin CS-50, Rheometric Scientific ARES, and Paar Physica MCR-300). Off-line rheometric measurements could not be made using cellulose fibre suspensions due to compression and drainage of the sample. For the surfactant solutions tested, the viscosity at the pipe wall decreased by an order of magnitude corresponding to an order of magnitude increase in the wall shear rate. In contrast, the viscosity at the pipe wall for the cellulose fibre suspensions decreased only slightly corresponding to the same increase in the wall shear rate. However, increasing the concentration of cellulose fibres from 1 to 3 % by weight increased the suspension viscosity at the pipe wall by a factor of about 2 to 3 depending on the shear rate. In general for a given throughput, as the concentration of the cellulose fibres increases, the suspension becomes stress, shear rate and shear viscosity distributions in the pipe, which are directly related to the more shear thinning as indicated by the decreasing values of the power-law exponent. The non-invasive in-line UVP-PD rheometric method also enabled the determination of the radial shear local microstructure (ie. the orientation) of particles in the suspension. In the absence of the present in-line method, it would not have been possible to study the rheological behaviour of non-Newtonian systems such as aqueous cellulose fibre suspensions even in conventional off-line rheometers.

1. INTRODUCTION

The rheology of industrial suspensions depends on the processing conditions, which affect the microstructure, and the shear and elongational flow fields. The product quality is usually controlled based on shear rate dependent viscosity information obtained using off-line rheometers as there exist no in-line rheometers for suspensions, which are usually opaque. This method is not reliable as its flow field could be significantly different to that in the actual flow process. In addition off-line rheometers cannot be used for in-line purpose because of the lack of suitability of their geometry and invasive nature. They are also difficult to use for suspensions with extreme rheological properties. Takeda (1-7) developed a pulsed ultrasound echographic method for the measurement of velocity profiles in flowing suspensions, which was expanded and manufactured as an Ultrasound Velocity Profile (UVP) Monitor by Met-Flow SA (8). Using this Monitor, Windhab and Ouriev (9-15) developed a system and a methodology for in-line rheometry of concentrated opaque suspensions by measuring simultaneously the velocity profile (UVP) and the pressure difference (PD) in a pipe section of a process. This was successfully tested on industrial and model suspension systems such as chocolate transport in pipes during chocolate and fat crystallisation processes (9-15), and starch in glucose syrup. The present paper investigates experimentally the above UVP-PD methodology for industrial surfactant solutions (shampoo) and model cellulose fibres in water suspensions using the UVP-PD in-line rheometer system. It also compares the shear rate dependent viscosities measured by UVP-PD in-line rheometric method with those measured by conventional off-line rheometers.

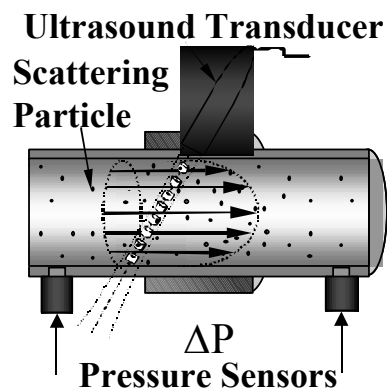


Figure 1. Flow adapter, ultrasound transducer and pressure sensors for the UVP-PD method.

2. THEORY

The principle of measurement of velocity profile by the Ultrasound Velocity Profile (UVP) Monitor is explained in detail by Takeda (1-7) and is summarised in the Met-Flow users guide (8). The in-line methodology developed by Windhab and Ouriev (9-15) involving the measurement of UVP and pressure difference (PD) to obtain the shear rate dependent viscosity using rheological models is explained by these authors and is discussed briefly here (see Fig.1 for working principle). The instrument measures instantaneous spatial velocity distribution in a flowing suspension along the axis of an emitted Pulsed Ultrasound beam by measuring the Doppler shift in the frequency of the reflected ultrasound and time delay. Equations for the radial velocity profiles during the flow of shear thinning (or thickening) and yield-stress suspensions in a pipe are presented below using respectively power-law and Herschel-Bulkley rheological models.

For laminar flow of a liquid of viscosity η in a pipe of radius R and length L , the variation with radius r in the shear stress is $\tau = r\Delta P / 2L$ (1) in which ΔP is the pressure drop, the wall shear stress being $\tau_w = R\Delta P / 2L$. If v is the velocity at any radius r , then the shear rate $\dot{\gamma} = -dv/dr$ and shear viscosity $\eta = \tau / \dot{\gamma}$. Power-law and Herschel-Bulkley rheological models are considered below to obtain the radial velocity, shear rate and shear viscosity for shear thinning (or thickening) fluids with and without an yield stress respectively.

2.1 Power-law model

The variation in shear stress τ with the shear rate $\dot{\gamma}$ for a power-law liquid is $\tau = K\dot{\gamma}^n$ (2) where K is the consistency index and n is the power-law exponent. Eqs.(1) and (2) can be combined and integrated (assuming zero velocity at pipe wall) to give the radial velocity, shear rate and viscosity profiles :

$$v = (nR/(1+n))(R\Delta P / 2LK)^{1/n} (1 - (r/R)^{1+1/n}) \quad (3); \quad \dot{\gamma} = (r\Delta P / 2LK)^{1/n} \quad (4)$$

$$\eta = \tau / \dot{\gamma} = K (r\Delta P / 2LK)^{1-1/n} \quad (5). \quad \text{At the pipe wall, the shear rate and viscosity are given}$$

$$\text{by } \dot{\gamma}_w = (R\Delta P / 2LK)^{1/n} \quad (6) \quad \text{and} \quad \eta_w = \tau_w / \dot{\gamma}_w = K (R\Delta P / 2LK)^{1-1/n} \quad (7).$$

Although Power-law model predicts (Eq.(5)) an unrealistic infinite shear viscosity at the centre of the pipe for shear thinning liquids ($n < 1$), it predicts a realistic finite viscosity at the pipe wall. Eq.(3) can be used to obtain the volume flow rate :

$$Q = 2\pi \int_0^R v r dr = \frac{\pi n R^3}{(3n+1)} \left(\frac{R\Delta P}{2LK} \right)^{1/n} \quad (8)$$

2.2 Herschel-Bulkley model

Some fluids behave like solids and do not flow until a critical yield stress τ_0 is exceeded. Application of stress is greater than the yield stress results in the formation of a moving plug in the centre of the pipe. For fluids with an yield stress and power-law behaviour above yield stress, Herschel Bulkley model can be used :

$\tau = \tau_0 + K\dot{\gamma}^n$ (9). This and Eq.(1) can be combined and integrated (assuming zero velocity at pipe wall) to give the radial velocity, shear rate and viscosity profiles :

$$v = (n/(1+n))(\Delta P / 2LK)^{1/n} ((R - R_*)^{1+1/n} - (r - R_*)^{1+1/n}) \quad (10)$$

$$\dot{\gamma} = (\Delta P / 2LK)^{1/n} (r - R_*)^{1/n} \quad (11); \quad \eta = \tau / \dot{\gamma} = K (\Delta P / 2LK)^{1-1/n} r / (r - R_*)^{1/n} \quad (12)$$

where $R_* = 2L\tau_0 / \Delta P$ is the plug radius. At the pipe wall, the shear rate and viscosity are

$$\text{given by } \dot{\gamma}_w = (\Delta P / 2LK)^{1/n} (R - R_*)^{1/n} \quad (13)$$

$$\text{and } \eta_w = \tau_w / \dot{\gamma}_w = K (\Delta P / 2LK)^{1-1/n} R / (R - R_*)^{1/n} \quad (14)$$

Herschel-Bulkley model also predicts (Eq.(12)) an unrealistic infinite shear viscosity at the centre of the pipe for both shear thinning and thickening liquids, it also predicts a realistic finite viscosity at the pipe wall.

$$Q = \pi v_* R_*^2 + \frac{\pi n R^2 (R - R_*)^{1+1/n}}{(n+1)} \left(\frac{\Delta P}{2LK} \right)^{1/n} \left[1 - \frac{2n}{(3n+1)} \left(1 - \frac{R_*}{R} \right)^2 - \frac{2nR_*}{(2n+1)R} \left(1 - \frac{R_*}{R} \right) - \left(\frac{R_*}{R} \right)^2 \right]$$

in which the plug velocity v_* can be obtained by setting $r = R_*$ in Eq.(10).

3. EXPERIMENTAL

3.1 Set-up

The present experimental set-up and procedure is described in detail by Wiklund and Johansson (2001) and is similar to that used by Ouriev (2000). However, a few relevant details are given here. The flow loop consists of a stainless steel tank with a propeller agitator, a positive displacement pump, a 23 mm diameter stainless steel pipe, a flow adapter made of a plastic composite PEEK30 fitted with a 1mm diameter 4 MHz frequency Ultrasound (U) Transducer in a 8 mm housing, a UVP Monitor (Met-Flow SA, Model UVP-X3-Psi, Switzerland), a Digital Oscilloscope (Yokogawa, Japan), two silicon diaphragm pressure sensors (PS), a PC for pressure and suspension weight data acquisition and a valve at the exit of the pipe to re-circulate the suspension.

3.2 Procedure

Prior to the flow experiments, a pulse of an ultrasound is generated in a measured sample of the suspension using a 4 MHz U transducer and UVP Monitor, the time difference for the reflected pulses being detected by the oscilloscope at two different vertical positions. The velocity of sound is then calculated using the distance between the two points measured by a digital height gauge (Tesa Brown and Sharpe SA, Switzerland). The tank is filled with the suspension, agitated mildly, the pump is set to the desired flow rate and the flow is allowed to attain the steady-state. The pressure difference in a pipe section of 1.16 m is measured by the PS connected to a PC (inverted manometer used for low pressure differences) which also simultaneously measures the weight of the suspension collected in a bucket put on a digital weighing balance in a given time. The Met-Flow UVP Monitor is triggered for measurement after setting the ultrasound parameters corresponding to the appropriate frequency and velocity of sound. Each raw velocity profile consists of Doppler frequency shift units at 128 points along the ultrasound beam, only less than about 30 points lying within the pipe diameter. 1024 such radial raw velocity profiles are recorded and stored on the hard disc of the in-built PC. These are then converted into velocity units along the pipe diameter allowing for the 20° angle of the ultrasound transducer with the normal to the pipe axis. Best velocity profiles are selected by comparing the measured volume flow rate with that obtained by integrating the velocity profiles. For comparison purposes, the shear rate dependent viscosities are measured using conventional off-line rotational rheometers such as Physica, ARES, Bohlin. Two industrial viscoelastic surfactant solutions and three cellulose fibres in water suspensions at three different weight percentages of fibres are investigated. However, off-line rheometers could not be used for fibre suspensions as fibres came out leaving mostly water in the cup.

4. RESULTS AND DISCUSSION

4.1 Surfactant solutions

The experimental (symbols) velocity profiles along the pipe diameter during the flow of viscoelastic surfactant (Shampoo AFi) solution (density $\rho = 1022 \text{ kg/m}^3$, velocity of sound $c = 1503 \text{ m/s}$) at different flow rates measured by the in-line UVP-PD method is shown in Fig.2. These are well represented by the theoretical profiles (curves) obtained by fitting the experimental data and pressure drop using the power-law model (Eq.(3)) with the values of constants n and K listed in the Table 1a. The flow is laminar since the maximum value of the

Reynolds number (Re_{max}) is less than 725 validating the use of the model equations. This is also true for shampoo XI ($\rho = 1012 \text{ kg/m}^3$, $c = 1518 \text{ m/s}$) for which $Re_{max} < 253$.

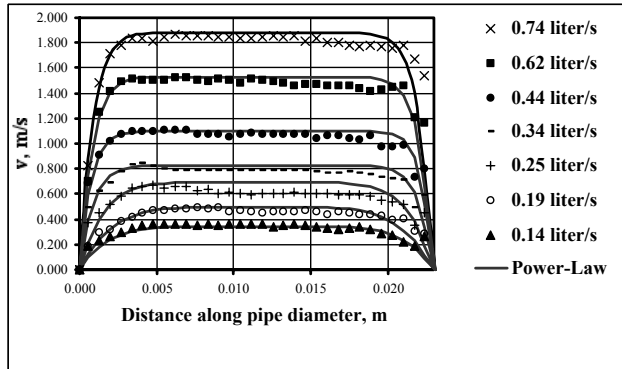


Fig.2. Experimental and Power-Law fitted velocity profiles for Shampoo AFi.

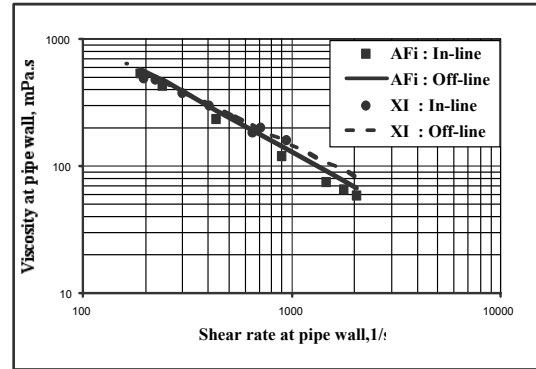


Fig.3. Shear rate dependent viscosities measured by in-line UVP-PD and off-line Physica rheometers.

Table 1. Power-Law model parameters obtained from experimental velocity profiles.

(a). Shampoo AFi

Flow rate, liter/s	Pressure drop, Pa	n	K
0.14	20680	0.19	37.7
0.19	20619	0.22	30.6
0.25	20500	0.16	38.3
0.34	21812	0.09	59.4
0.44	22330	0.07	66.0
0.62	23433	0.08	62.9
0.74	24012	0.09	60.9

(b). Shampoo XI

Flow rate, liter/s	Pressure drop, Pa	n	K
0.14	19499	0.28	22.4
0.19	21528	0.30	21.2
0.25	22495	0.28	22.6
0.33	24306	0.26	25.3
0.43	24033	0.19	33.9
0.61	29013	0.24	29.8
0.72	30870	0.19	41.1

The variation in the viscosity η_w with shear rate $\dot{\gamma}_w$ at the pipe wall obtained using the in-line UVP-PD method (symbols) and Eqs.(7) and (6) with the values of the power-law constants n and K for the data shown in Figure 2 and for shampoo XI, is shown in Figure 3. This agrees well with the variation in viscosity with shear rate measured by off-line Paar Physica MCR-300 rheometer are also shown by the full lines for AFi (n = 0.09, K = 69.4) and broken lines for XI (n = 0.19, K = 39.2) shampoos respectively, the off-line power-law constants corresponding to the whole range of shear rates encountered in the in-line experiments. Other off-line rheometers such as Rheometric Scientific ARES and Bohlin CS-50 are also used to measure the shear rate dependent viscosity. However, these could not be used at higher shear rates as the highly shear thinning shampoos started leaving the Couette geometry.

4.2 Aqueous cellulose fibre suspensions

Figure 4 shows the experimental (symbols) velocity profiles along the pipe diameter during the flow of 2% by weight of cellulose fibres in water suspension at different flow rates measured by the in-line UVP-PD method. Once again these are well represented by the theoretical curves obtained by fitting the experimental data and pressure drop using the power-law model (Eq.(3)) with the values of constants n and K listed in the figure. Similar agreement is obtained using Herschel-Bulkley model (eq.(10)). This is also true for cellulose

concentrations of 1% and 3%. The flow is once again laminar since the maximum value of the Reynolds number (Re_{max}) is less than 504 validating the use of the model equations.

Figure 5 shows the variation in the viscosity with the shear rate at the pipe wall obtained using the measured velocity profiles using Power-law (Filled symbols) and Herschel-Bulkley (open symbols) for 1%, 2% and 3 % cellulose in water. Both the models seem to predict about the same viscosity for a given shear rate at the pipe wall, the agreement between them depending on the accuracy of the plug radius R_* assumed. In general the cellulose fibre suspensions are shear thinning as indicated by the decrease in the viscosity at the pipe wall with the shear rate. The viscosity of 3% cellulose fibre suspension is about thrice that of the 1% suspension at low shear rates, the factor being about 2 at high shear rates. However, off-line rheometers could not be used to measure the shear rate dependent viscosity as the fibres got separated from the water in the suspension during measurement.

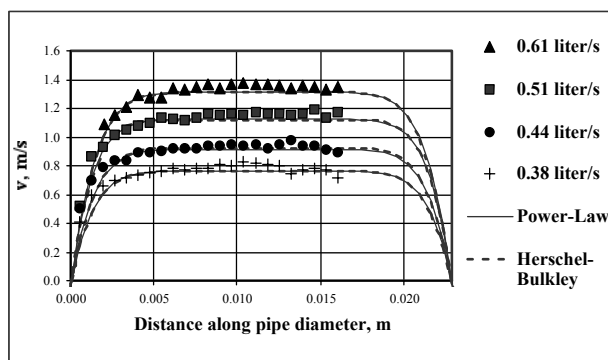


Fig.4. Experimental and fitted velocity profiles obtained by in-line UVP-PD method for 2% cellulose fibre in water suspension .

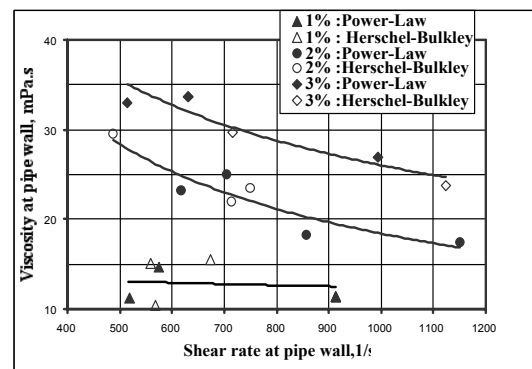


Fig.5. Variation in viscosity with shear rate at pipe wall determined by in-line UVP-PD method for different % cellulose fibre in water suspensions.

Table 2. Power-Law and Herschel-Bulkley model parameters obtained from experimental velocity profiles for 2% cellulose in water suspensions ($\rho = 1000 \text{ kg/m}^3$, $c = 1502 \text{ m/s}$).

Flow rate, liter/s	Pressure drop, Pa	Power-Law		Herschel-Bulkley		
		n	K	n	K	R_* mm
0.38	2916	0.13	6.24	0.24	2.12	4
0.44	3164	0.13	6.49	0.24	2.10	4
0.51	3564	0.13	7.50	0.19	3.69	3
0.61	4076	0.14	7.50	0.27	2.19	4

For each of the measured velocity profiles in Figures 2 and 4, the shear rate decreases along the pipe diameter from a maximum value at the pipe wall to zero at the centre of the pipe. This corresponds to an increase in viscosity along the pipe diameter from a minimum value (corresponding to wall shear rate) to a constant maximum value at the centre of the pipe for the shear thinning Shampoo solution and cellulose in water suspensions. However, both Power-law and Herschel-Bulkley models predict an unrealistic infinite viscosity at the centre of the pipe where the shear rate is zero. The volume flow rates obtained by integrated velocity profiles represented by Power-law or Herschel-Bulkley model and pressure measurement by UVP-PD method has an error of 10 to 30% compared with those measured by the gravimetric method ; the error decreased with the flow rate as the accuracy of pressure drop measurement

increased. The accuracy also increased with the extent of shear thinning as reflected by the Shampoo solutions. The deviation could also be due to the fact that the consistency index K and exponent n may not be constant over the entire range of shear rates encountered along the radius of the pipe.

5. CONCLUSIONS

The in-line UVP-PD methodology developed by Windhab and Ouriev (9-15) using the Met-Flow UVP Monitor is successfully tested to obtain shear rate dependent viscosities of shear thinning viscoelastic surfactant shampoo solutions and aqueous cellulose fibre suspensions. These agreed well with those obtained using conventional off-line Paar Physica rheometer for shampoo solutions. The rheological behaviour of cellulose fibre suspensions could not be measured using the off-line rheometers due to compression and drainage of the sample, thereby making the in-line UVP-PD method as the only alternative method.

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