Laboratory-Scale Pipe Rheometry: A Study of a Microfibrillated Cellulose Suspension

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In this work a novel laboratory-scale pipe rheometer is utilized in rheological characterization of concentrated MFC and cellulose suspension. The method is based on a combination of pulsed ultrasound velocity profiling (UVP) and pressure difference measurement (PD). The rheological properties of the suspensions are described in terms of viscosity and pressure loss. The results are compared to rotational rheometer results. It is demonstrated that well controlled pipe flow environment together with UVP-PD technique is efficient in characterizing the rheological flow behavior of complex slurries.

Keywords: inline rheology, ultrasonic velocity profiling, microfibrillated cellulose

1 INTRODUCTION

Ultrasound velocity profiling (UVP) has been accepted as an important measuring technique in fluid dynamics and engineering applications. Furthermore, UVP is increasingly being used together with pressure difference measurement as a non-invasive rheological measuring technique commonly known as UVP-PD. This non-invasive method is capable of in-line rheological measurements for concentrated, opaque fluids in real-time [1-5].

Cellulose fibers generally form an opaque flocculated suspension with high viscosity already at very low mass concentrations. In many cases conventional rheometers fail to produce reliable information for these suspensions. Recently, both UVP and UVP-PD methods have been successfully applied in probing details of cellulose fiber suspension flows and their rheological properties [6-9].

Recent developments in cellulose fiber disintegration have facilitated production of micro- and nanofibrillated cellulose (MFC/NFC). The properties of these fibrils differ significantly from cellulose fibers, especially in specific surface area, aspect ratio, strength and flexibility [10]. Development of new materials and applications utilizing MFC/NFC requires understanding the rheology of them.

The aim of this work was to develop a laboratory-scale rheometer which has minimal restrictions in sample properties (consistency, particle dimensions) and which enables measurements in varying shear conditions. Tube flow geometry was selected due to its practical significance in process industry and effortless modification possibilities. The UVP-PD method was preferred to other ones due to its applicability for opaque fluids. Furthermore, UVP-PD is based on direct measurements and includes no assumptions concerning the flow pattern or boundary conditions.

The pipe rheometer was firstly applied for studying rheological properties of a commercial MFC and cellulose suspensions in high consistency. The rheological properties of the suspensions are described in terms of viscosity and pressure loss behavior. The viscosity results are compared to rotational rheometer results.

2 MATERIALS

Microfibrillated cellulose was a commercial product Celish® KY-100G (Daicel Chemical Industries, Japan) made from purified wood pulp. The average length and diameter of Celish® fibers reported by Tatsumi et al. [11] are 350 µm and 15 µm respectively.

As a reference for MFC, bleached birch cellulose was measured at similar conditions as MFC pulp. The approximate average fiber length and width are 0.9 mm and 20 µm, respectively.

Before measurements, the cellulose fibers were dispersed in deionised water and disintegrated. The mass consistency of the slurries was 1.5 % and temperature 20±0.5 °C.

3 METHODS

3.1 Laboratory-scale pipe rheometer

The schematic of the laboratory-scale pipe rheometer is shown in Fig 1. The pipe rheometer consists of two chambers connected by a replaceable vertical pipe section. The volume of each chamber is approx. 3 liters. The measurement pipe used in this study was a smooth acrylic tube with inner diameter of 16 mm.

The measurement is initiated by filling the sample

fluid in the bottom chamber. The fluid is then pumped to the upper chamber. The desired filling level is determined by an ultrasound surface detector. The measurement is started by opening the valve downstream the measurement tube. The flow in the measurement tube can be driven by sole gravity or by overpressure in the upper chamber. The pressure driving the flow is controlled via a regulator and a pressure level sensor. Therefore the flow in the measurement tube is undisturbed by e.g. pulsations from pumps.

The mass flow rate of the fluid is determined with three weight sensors and the volume flow rate with the ultrasound surface detector. With these two independent measurements also the density of the fluid can be determined. The pressure difference in the measurement tube is measured over a distance of 0.99 m.

Rheological characterization of the fluid is carried out utilizing the UVP-PD method. Region of steady flow is determined from the measurement data and analyzed for mean pressure loss, mass rate and volumetric flow rate. The result is combined with the simultaneously measured mean velocity profile.

The operation of the pipe rheometer is fully automated starting from sample loading to execution of user-defined measurement scheme and sample removal. The measurement scheme can cover various processing conditions in the same measuring period.

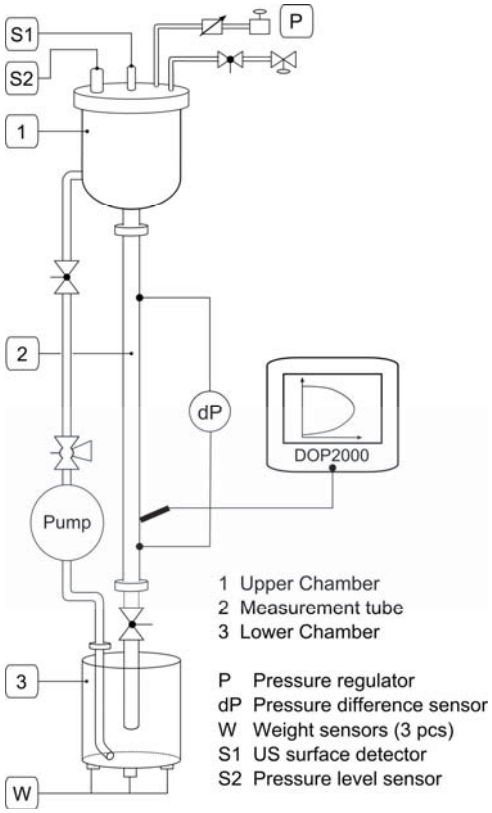

Figure 1: Schematic of the laboratory-scale pipe rheometer.

3.2 Ultrasound velocity profiling

The velocity profiles are measured using a pulsed UVP (DOP2000, Signal-Processing S.A., Switzerland). The settings used for the velocity measurements are summarized in Tab. 1. Depending on the flow speed 100 – 1000 individual profiles were recorded and analyzed for each measurement.

3.3 Reference measurements with a conventional rheometer

The viscosity of the MFC dispersion was measured at 20 °C with a stress controlled rotational rheometer (AR-G2, TA Instruments) equipped with the vane geometry. The diameter of the cylindrical sample cup was 30 mm and that of the vane 28 mm. The length of the vane was 42 mm.

After loading the sample to the measuring geometry, it was allowed to rest for 5 min before the measurement was started. The viscosity was measured in strain controlled mode with a gradually increasing shear rate in the range of 1E-4 - 100 s^{-1}. The reported viscosity at a certain shear rate was taken after a constant shear stress was reached or after a maximum time of 2 min.

3 RESULTS

3.1 Pipe rheometer

The experimental results acquired from the pipe rheometer can be analyzed using different approaches including implementations of rheological models as e.g. described by Wiklund et al. [4]. In this work the results are presented based on the direct measurements. Firstly, the results are analyzed utilizing only the pressure loss and volumetric flow rate data assuming laminar parabolic velocity profile. For a laminar Newtonian flow in a straight tube the apparent shear rate $\dot{\gamma}_a$ and shear stress τ_W at the tube wall are given by

$$\dot{\gamma}_a = \frac{4Q}{\pi R^3} \tag{1}$$

$$\tau_W = \frac{\Delta P R}{2L} \tag{2}$$

where Q is the volumetric flow rate, R is the radius of the tube and ΔP is the pressure difference over the length L of the tube. The shear viscosity is the ratio of shear stress to shear rate, $\eta_a = \tau_W / \dot{\gamma}_a$.

The first analysis corresponds to a conventional measurement of viscosity using e.g. capillary viscometer. In this analysis no corrections due to non-Newtonian profile of slip are applied. The result is referred as apparent viscosity.

The second analysis utilizes the experimental mean velocity profile and thereby gives viscosity

measured locally in the flow. This method includes no assumptions concerning the flow profile or boundary condition. The local shear rate $\dot{\gamma}(r)$ is obtained directly from the velocity profile. As the shear stress at each distance from the tube wall is

$$\tau(r) = \tau_W (1 - r/R), \quad (3)$$

the shear viscosity can be calculated locally. This result is referred as intrinsic viscosity $\eta(r) = \tau(r)/\dot{\gamma}(r)$.

The pressure drop as a function of mean flow velocity is shown in Fig. 2. For the birch cellulose suspension typical plug flow and lubrication layer regimes can be found as reported e.g. by Jäsberg [7]. Velocity profiles for selected flow rates are shown in Fig 3 (Roman numbers correspond to different flow rates).

In addition to the substantial difference in the pressure loss magnitude, the qualitative pressure loss behavior of MFC suspension differs clearly from that of cellulose suspension. Three different flow regimes can be identified by inspecting the pressure loss for increasing velocity. At low flow rates the pressure drop increases almost linearly and reaches a maximum value at velocity of approx. 0.4 m/s. After the maximum, the pressure loss decreases abruptly and then starts to increase linearly with velocity. All the velocity profiles (See Fig 4.) indicate clear slip at the wall. At low velocities, the shape of the profile is plug-like. As the velocity increases the plug starts to break down.

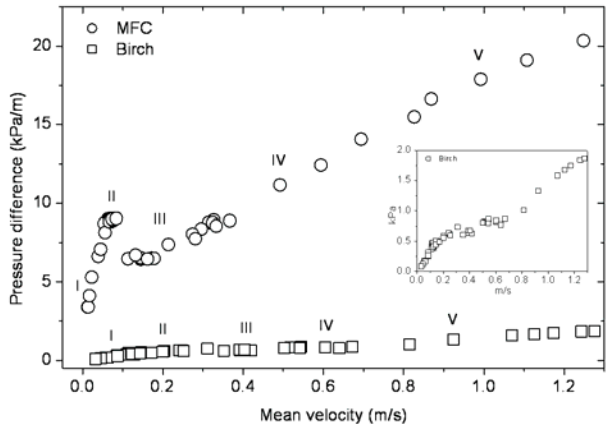

Figure 2: Pressure drop as a function of mean velocity. The roman numbers correspond to the velocity profiles in Figs 3 and 4.

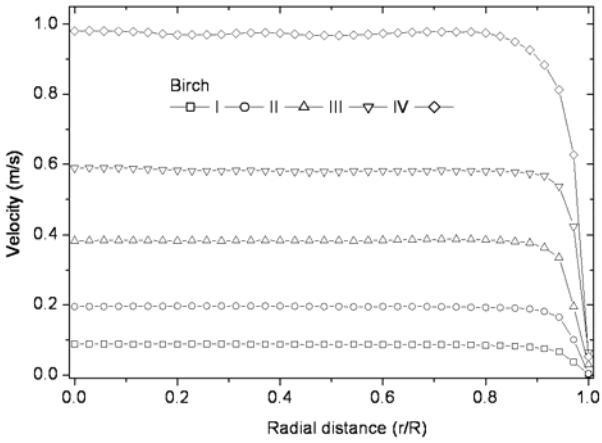

Figure 3: Mean velocity profiles for birch cellulose. The roman numbers for different profiles correspond to the pressure losses marked in Fig. 2.

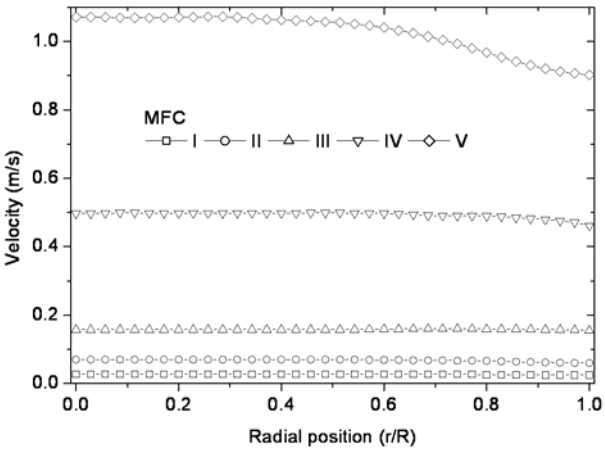

Figure 4: Mean velocity profiles for MFC. The roman numbers for different profiles correspond to the pressure losses marked in Fig. 2.

The viscosity results are presented in Fig. 5. As expected from the pressure loss results, the viscosity of MFC is clearly higher than that of birch cellulose. The results obtained from UVP-PD and rotational rheometer agree well. For both suspensions, the viscosity is strongly dependent on the shear rate in the measured range of shear rates. Also the apparent viscosity agrees qualitatively with the other results. Due to non parabolic velocity profile the average shear rate for apparent viscosity is too high, especially for low flow rates. The Reynolds number determined using the apparent viscosity values range from 1 to 150 for the MFC and from 30 to 1300 for the birch cellulose.

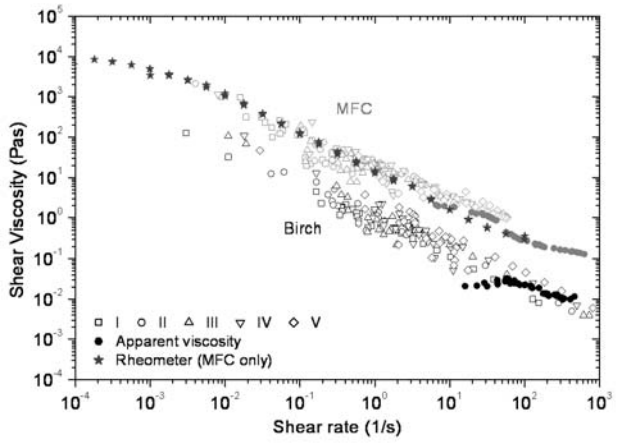

Figure 5: Intrinsic (open symbols) and apparent (solid symbols) shear viscosities for birch cellulose (black) and MFC (gray).

4 CONCLUSIONS

The pipe rheometer introduced in this study facilitates rheological studies for highly-concentrated, complex slurries in process like environment. The rheological measurements are based on the UVP-PD method. The results are highly consistent with the data from rotational rheometer.

Table 1: Main UVP settings

Setting	Value
Sensor base frequency	8 MHz
Sensor active diameter	5 mm
Doppler angle	80°
PRF	1–2.7 kHz
Burst length	4 cycles

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