RHEOLOGICAL CHARACTERIZATION OF MICRO-FIBRILLATED CELLULOSE FIBRE SUSPENSION USING MULTI SCALE VELOCITY PROFILE MEASUREMENTS

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ABSTRACT

A rheometric method based on velocity profiling simultaneously by optical coherence tomography and the ultrasound velocity profilometry was introduced and used in a preliminary study of the rheological and boundary layer flow properties of microfibrillated cellulose. The two velocity profiling methods appear adequate and complementary for rheological characterization of opaque complex fluids. The ultrasound method is useful in measuring the velocity profile in the interior parts of the tube, while the optical technique is capable of high-resolution measurement of the boundary layer flow close to the tube wall.

The preliminary results obtained for a 0.4% micro-fibrillated cellulose suspension show typical shear thinning behaviour in the interior part of the tube while the near wall behaviour shows existence of a slip layer of thickness ~200 µm. Both the velocity profile measurement and the imaging mode data obtained by the optical coherence...
tomographic method indicate that the slip layer is related to a concentration gradient appearing near the tube wall. In a sublayer of thickness ~100 μm, the fluid appears nearly Newtonian, and the viscosity value approaches that of pure water with decreasing distance from the wall.

INTRODUCTION

Pulp fibre wall consists of a natural composite material comprised of cellulose microfibrils. Lately, there has been growing interest towards disintegrating the fibre structure to its elementary fibrils, thereby producing natural raw stock for novel materials. Disintegrating can be done using a combination of mechanical grinders and chemical and enzymatic treatments that typically yield aquatic suspension of fibrils. The produced fibrils, called micro (nano) fibrillated cellulose fibres (MFC), are typically of length 100–1000 nm and diameter 5–30 nm [1]. Both the mechanical and the rheological properties of these fibres and fibre suspensions are quite different from e.g. those of wood fibres, and as yet, relatively poorly known.

The rheological and flow properties of natural and synthetic fibre suspensions are diverse, and depend strongly on fibre properties and fibre mass concentration. The overall characteristics of suspensions of relatively simple fibres can, to some extent, be classified in terms of Crowding number Cr that depends on concentration, dimensions and material density of fibres, and correlates with the mean number of contacts between fibres in the suspension [2–5]. Such a classification is not applicable in MFC suspension which tends to form a gel showing yield stress, thixotropy and shear thinning properties [6–8] already at very low concentration. Iotti et. al. [6] reported also hysteresis and peculiar time dependent behaviour of MFC shear viscosity using parallel plate rheometer geometry. An important and well known problem in interpreting the data obtained from conventional rheometric experiments for complex fluids is that the measured result depends not only on the actual ‘bulk’ rheological properties, but also on the sometimes intricate boundary layer behaviour of the flow in the particular device being used. It is not always straightforward to extract and correct for the boundary effects, such as wall slip, to obtain the desired rheological properties. Another related issue leading to similar uncertainties is that in many conventional rheological techniques, the data analysis is based on an assumed velocity profile. Often, no practical means are available for verifying whether the assumed profile is actually realized in the measurement or not.

Rheometric methods utilizing velocity profiling are based on measuring the actual flow velocity profile and wall shear stress simultaneously. The techniques applicable especially for optically turbid media are typically based on Nuclear
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Magnetic Resonance Imaging (NMRI) [9,10] or Ultrasound-Doppler Velocity Profilometry (UVP) [11–16]. Both UVP and NMRI have been tested for several rheologically complex fluid and multiphase systems of practical interest in e.g. in food, paper and chemical industry. The concept is well established and has been implemented as in-line rheometers in industrial processes providing means for process monitoring and quality control [9–15].

Velocity profiling with UVP and NMRI allows replacing an assumed velocity field by an actually measured profile in analysing the data for rheological properties of the fluid. However, due to their limited spatial resolution and, especially for UVP, disturbance caused by the tube wall – fluid interface [17–19], these methods are hardly accurate enough to resolve the flow profile in the immediate vicinity of the tube wall, where e.g. apparent slip can occur. Optical Coherence Tomography (OCT) is a non-invasive technique capable of fast real-time high-resolution imaging of the internal structure of an opaque scattering medium in the vicinity of its surface. The imaging depth of OCT is limited by the attenuation of light in the material, and can be up to 2.5 mm in air. In addition to giving access of structural data, the method can provide velocity information. Furthermore, the OCT method appears capable of accurate high-resolution measurement of velocity profile very close to a channel wall, and is thus well suited in detailed study of the boundary layer flow behaviour of complex fluids with appropriate optical properties [20].

In this work we report preliminary results on rheological and boundary layer flow properties of a micro-fibrillated cellulose suspension, obtained by a novel in-line rheological method utilizing velocity profiling. The velocity profiles are measured by combining two complementary methods, the optical coherence tomography and the ultrasound velocity profilometry. Here, OCT is used to measure the velocity profile in the immediate vicinity of a transparent tube wall (typically closer than 0.5 mm), while UVP provides the same information in the interior parts of the tube.

EXPERIMENTAL SET-UP

The experiments were done in a 1500 mm long and 8.6 mm inner diameter optical grade glass pipe with 2.5 mm wall thickness. For the OCT measurement (located 113 diameters from the inlet), a flat surface was ground in the pipe outer surface to minimize the glass thickness and to prevent unwanted refraction at the outer surface. The flow was driven by gravity or pressurized air to allow for a steady, pulsation-free flow. The mass flow rate was obtained with a laboratory scale.§

§ Sartorius TE3102S
The wall shear stress at each flow rate was found based on pressure difference measurement between two 1.5 mm diameter taps drilled through the pipe wall 1000 mm apart (the first measurement point 52 diameters from the inlet). The measurement was done using calibrated pressure difference sensors. Preparation of the MFC suspension was done by diluting the 10% furnish in deionized water under high intensity mixing. Mixing period was 30 minutes. For OCT velocity profile measurements 1.2% by volume of low-fat UHT milk was added as light scattering tracer. Structure measurements were done without added milk. Imaging frequencies (axial scan rates) of 5.5, 28 and 91 kHz were used depending on the velocity. Each slice image consisted of 1000 axial scans equaling physical width of 1.00 mm. For each measurement 200 slice images were recorded and analysed for mean velocity profile. Doppler angle was 5 deg.

The microfibrillated cellulose used in this study was a commercial product Celish® KY-100G made from purified wood pulp. The average length and width of fibers are 350 μm and 15 μm respectively [21]. The fibre surfaces are, however, very strongly fibrillated. The size distribution of these fibrils is very wide ranging from microscale to nanoscale.

**Ultrasound Velocity Profiling (UVP)**

Ultrasound velocity profiling technique is based on using an emitter/receiver to send a series of short ultrasound bursts into the flow, and detecting the sound reflected from the target particles moving along with the flow. The location of the particles is acquired with the time-of-flight method using the known velocity of

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2 Rosemount, sensor types 3051 and 2051
3 Daicel Chemical Industries, Japan
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sound. The velocity of the moving particles is calculated from the cross-correlation between the echoes from consecutive pulses. Thus, UVP measures the velocity component in the direction of the ultrasound beam. In the present study, a DOP2000® device equipped with an 8 MHz ultrasound probe was used. The angle between the probe axis and the pipe wall was 74°. The length of a single ultrasound pulse was set to 2 cycles corresponding to a pulse length of ~ 0.375 mm in water. The ultrasound pulse repetition frequency was varied between 500 and 1000 Hz in order to exploit the full velocity resolution range. With the used UVP device, the velocity data acquisition is done with 8 bit dynamics. In all measurements, 8 consecutive pulse emissions were used to calculate a single velocity value in each sampled depth locations. A series of 150 – 500 individual velocity profiles was recorded for each flow rate, and the final mean velocity profile was calculated as an average of these.

Optical Coherence Tomography (OCT)

The spectral domain optical coherence tomography, used in this work, is a fusion of laser Doppler velocimetry and optical coherence tomography. A standard OCT setup includes a low-time coherence light source, such as a superluminescent diode (SLD), and a Michelson interferometer. The interferometric technique provides high dynamic range, spatial resolution and sensitivity, which makes OCT useful for analysing the internal structure of many heterogeneous materials of interest within e.g. biomedical and materials research.

The OCT device used in this study was Telesto Spectral Domain OCT®. The central wavelength of the SLD used in the device is 1325 nm with bandwidth in excess of 150 nm. The resolution in the axial (beam) direction in water is better than 5 μm. High axial resolution is possible because interference is observed only when the optical path lengths of light in the target and reference arms match to within the source coherence length. The lateral resolution was 15 μm in the present case.

The Fourier transform of the interferogram acquired with OCT imaging produces a complex signal [I(z) + iQ(z)]. The magnitude of the signal is used to create the structural OCT image. The complex part of the signal contains information on the phase of the interferogram. Changes in phase between consecutively acquired interferograms can be attributed to a Doppler frequency shift induced by particle motion. Doppler frequency shift is calculated by spatially averaging phase shifts within a sliding 2D window and using a Kasai autocorrelation function [22].

® Signal-Processing S.A., Switzerland
® Thorlabs, USA
Test Case: Flow of water in a tube

In order to demonstrate the techniques based on combining the velocity data from OCT and UVP, we first apply it to a test case for which the velocity profile is well known: the turbulent flow of water in a straight tube with circular cross-section and smooth walls. In that specific case, the velocity profile in the near-wall inner layer is accurately described by the Spalding scaling law formulated in terms of the dimensionless distance from the tube wall, \( y^+ = u^+y/v \), and the dimensionless flow velocity \( u^+ = u/u^* \), where, \( y \) is the distance from the wall, \( u^* = \sqrt{\tau_w/\rho} \) is the friction velocity, \( \tau_w \) is the shear stress at the wall, \( \nu \) is the kinematic viscosity of water [23]. The Spalding profile (see Fig. 3 b) smoothly interpolates between the linear law of the wall, \( u^+ = y^+ \), and the logarithmic law \( u^+ = \frac{1}{\kappa} \ln y^+ + B \), where \( \kappa \approx 0.40 \) and \( B \approx 5.5 \) are constants. The linear and logarithmic laws are valid in the viscous sublayer at \( y^+ \lesssim 5 \) and in the overlap region \( y^+ \gtrsim 30 \), respectively. In the outer region, roughly at \( y^+ \gtrsim 300 \), the velocity profile deviates slightly upwards from the logarithmic law.

Warm tap water was cooled down to room temperature overnight for reducing the gas content of the water. During the measurements the water temperature...
was between 22 and 23 °C. For OCT velocity profile measurements 1.2% by volume of low-fat UHT milk was added as light scattering tracer. For UDV measurements a small amount of neutrally buoyant hollow glass spheres with 5 μm diameter was added to the water.

The measured results together with the standard profile correlations are shown in Figure 3. Both measured profiles quite accurately follow the scaling law. The UVP results extend in the overlap and outer layers, whereas the OCT results cover the viscous sublayer and part of the buffer layer between the viscous and overlap layers. Especially, the data from OCT measurement follows the theoretical profile with remarkably good accuracy over more than two orders of magnitude in $y^+$. Notice, that the OCT results shown include data also from laminar flows at relatively low Reynolds numbers. The linear law of the wall is, nevertheless, valid also for laminar flows. The spatial measurement range of the two methods is non-overlapping. In the present case, reliable data from OCT could be obtained in the distance range from 5 to 500 μm where the lower limit is set by the spatial resolution of the method, and the upper limit by attenuation of light in the slightly opaque fluid. Correspondingly, the practical measurement range for UVP includes the interior part of the tube, except of distances closer than about 1 mm from the walls. Closer to that distance, disturbances caused by sound reflected

![Figure 3](image-url)

**Figure 3.** Velocity profiles of water measured with OCT (circles) and UVP (squares) in dimensionless variables $y^+$ and $u^+$ plotted in linear scales a) and with logarithmic $y^+$ scale b). The dash-dotted line indicates the linear viscous sublayer profile and the dashed line is the logarithmic buffer layer profile. The solid line represents the Spalding inner layer correlation.
from the interface between the fluid and the tube wall, cause noticeable systematic error. In general, the practical measuring distance for both methods depends on properties of the tube wall and the fluid, and on device settings.

RESULTS
Velocity profiles
In Figure 4 shown is a set of flow velocity profiles of 0.4% MFC in a tube of diameter 8.6 mm, as measured with OCT and UVP techniques (experimental conditions are shown in Table 1). The profiles are obtained by a sequence of measurements with increasing flow rate. The flow condition is laminar in all cases. The useful measuring range of the two methods is non-overlapping. The overall behaviour of the velocity profiles measured with OCT and UPV appears consistent, and it seems possible to construct a plausible estimate of the entire profile by interpolating the velocity values through the rather narrow gap between the measured profiles. However, at this point we concentrate on analysing the data in regions actually measured.

The overall velocity profile appears to consist of two dynamically different parts. In the central region, at the distance range $200 \mu m \leq y \leq R$, the profile is relatively shallow and qualitatively resembles that of a pseudoplastic fluid. In

![Figure 4](image_url)

**Figure 4.** a) Examples of velocity profiles of MFC measured simultaneously with OCT (circles) and UVP (squares) as a function of the distance $y$ from the tube wall. The thick dashed vertical line gives the position of the centre-line of the tube. The gap between the thin dashed lines indicates the domain where accurate data is not available from the two measurement techniques. b) The near-wall velocity profiles measured by OCT (circles) together with fitted curves according to Eqn. (1) (dashed lines) and interpretation of various fitting parameters. The measurements have been done for increasing flow rate.
Table 1. Experimental conditions

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macroscopic scales the flow behaviour of MFC shown in Figure 4 a) appears as that of a shear thinning fluid with wall slip. The high-resolution OCT data reveals, however, that in a narrow near-wall region, the velocity profile is very steep and approaches rapidly zero with decreasing distance from the wall with no actual wall slip. The (apparent) wall slip is resolved as an exponentially growing velocity deficit in a narrow slip layer of thickness ~200 μm.

Indeed, within the region $y \leq 500$ μm covered by the OCT measurement, the velocity profiles can be accurately fitted by the formula

$$u(y) = \gamma_w^a y + u_s \left(1 - e^{-\frac{y}{\lambda_w}}\right),$$  \hspace{1cm} (1)

where $\gamma_w^a$, $u_s$ and $\lambda_w$ are free parameters. The wall layer velocity profiles together with fits according to Eqn. (2) are shown in Figure 4 b). Also shown is the graphical interpretation of various fitting parameters for an individual profile: $\gamma_w^a$ is the apparent shear rate at wall, $u_s$ is the (apparent) slip velocity and $\lambda_w$ is the characteristic thickness scale of the slip layer. The shear rate at the wall is given in terms of the primary fitting parameters by

$$\gamma_w = \left(\frac{du}{dy}\right)_{y=0} = \gamma_w^c + u_s / \lambda_w.$$  \hspace{1cm} (2)

Near-wall characteristics

The rheology of the MFC at the wall region can be characterized by considering the correlation between the measured wall shear stress $\tau_w$ and wall shear rate. Given the fitted parameters introduced in Eqns. (1) and (2), two different approaches can be taken by considering correlation of the wall shear stress either with the apparent shear rate $\gamma_w^a$ or with the actual wall shear rate $\gamma_w$. Figure 5 a) shows the measured wall shear stress plotted as a function of the apparent wall shear rate $\gamma_w^a$.
shear rate. The profile closely follows a 1/2 power law curve (dashed curve) indicating apparent shear thinning behaviour. Such behaviour is generally observed also in conventional rheometric measurements for MFC [6,7,8]. Instead, the correlation between wall shear stress and wall shear rate, plotted in Figure 5 b), show qualitatively different behaviour. Here, the data indicates close to ideal Bingham plasticity with yield stress ~0.3 Pa, and a constant slope practically identical to viscosity of water (dashed line).

A possible explanation for the results shown in Figure 5 is provided by assuming that at small shear stress region, \( \tau_w < 0.3 \) Pa, no slip layer exists, and the bulk properties of the fluid prevail up to the wall. When the wall stress exceeds a critical value, a slip layer is formed. This is also supported by the result shown in Figure 5 c) where the slip velocity appears to approach zero at a finite value of \( \tau_w \).

A plausible reason for formation of the slip layer is a near-wall hydrodynamical lift force causing the polymer particles to migrate away from the wall [24,25]. This leads to a small or vanishing value of MFC concentration at the wall, and to an increase of concentration with distance from the wall until it reaches its bulk value outside the slip layer. Such a phenomenon is well known to cause apparent wall slip in flows of particulate suspensions [26]. Direct evidence of such a mechanism in the present case is readily obtained by utilizing also the high-resolution imaging modality of OCT.

In Figure 6 shown are an instantaneous and an averaged OCT image of MFC suspension flowing near a tube wall. The OCT images are constructed as the spatial distribution of back-scattering index of light. The scattering index, in turn,
Correlates with the concentration of suspended particles that contribute to scattering. The light shades of grey appearing near the tube wall thereby indicate lower average concentration as compared to regions at larger distances. In addition, the grey-scale value saturates to a nearly constant value roughly at the distance 200 \( \mu m \) from the wall.

**Viscosity measurement based on velocity profiling**

The measured velocity profile and wall shear stress data allow us to calculate values of viscosity locally inside the tube as \( \mu(y) = \tau(y)/\dot{\gamma}(y) \), where \( \tau(y) = \tau_w (1 - y/R) \) is the shear stress at \( y \), and \( \dot{\gamma}(y) \) is the local shear rate given by the measured velocity profile. Each measured velocity profile thus yields values of viscosity in a range of shear rates present in the profile. Figure 7a) shows the local viscosity values as a function of local shear rate for a range of \( \tau_w \) rates utilizing the velocity profile data from both OCT and UVP measurements. Here, the local shear rate values are estimated by a straightforward interpolation of the velocity data. Notice, that the experimental profiles are already time averaged by the measurement settings and the primary data analysis, and thus smooth enough to allow for differentiation without introducing excess scatter of shear rate values. The data obtained from the inner parts of the tube by the UVP measurement again show shear thinning behaviour in the shear rate region \( \dot{\gamma} \lesssim 100 \) 1/s. The result is very similar to that found for MFC suspension by rotational rheometric techniques [6,8]. The near-wall behaviour is again qualitatively different. In the shear rates \( \dot{\gamma} \gtrsim 200 \) 1/s, viscosity appears nearly independent on shear rate. Notice that the data within each of the nearly horizontal groups of points observable at the high shear
rate comes from the same distance from the wall. The viscosity in the slip layer thus appears to correlate with the distance from the wall rather than shear rate. This is supported by Figure 7 b) where the viscosity calculated from the near-wall velocity profile is shown as a function of position for the same set of OCT data shown in Figure 7 a). In the region \( y < \lambda_n \) (~50–100 µm in the present case), viscosity indeed seems to depend only on distance from the wall, and approaches the viscosity of water with decreasing \( y \). Outside the slip layer, the correlation between viscosity and distance disappears (and is gradually replaced by correlation with shear rate). Obviously, the observed near-wall position dependence of viscosity arises due to MFC concentration gradient in that region. The concentration close to the wall is low enough such that even though the viscosity increases with concentration/distance, the fluid appears Newtonian in the region \( y \geq \lambda_n \).

**DISCUSSION**

We have introduced a novel rheological experimental method based on velocity profiling simultaneously by two techniques of different operating principle, the optical coherence tomography (OCT) and the ultrasound velocity profilometry.

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**Figure 7.** a) Viscosity of MFC suspension as a function of shear rate calculated locally from velocity profiles measured near the wall by OCT (circles) and inner parts of the tube by UVP (squares). Included is the profile data at \( y \leq 2.5 \) mm for various flow rates corresponding to wall shear stress range 0.8 – 3.0 Pa. b) Viscosity of MFC as a function of distance from the wall in the near-wall region calculated locally from velocity profiles measured by OCT. For clarity the spatial resolution is decreased in these figures.
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(UVP). The method was demonstrated in a study of micro-fibrillated cellulose (MFC) suspension flow in a straight tube. The two methods appear complementary as the UVP method is useful in measuring the velocity profile in the interior parts of the tube with relatively low, yet adequate spatial resolution, while the OCT techniques is capable of high-resolution measurement of the boundary layer flow very close to the tube wall.

The preliminary results for 0.4% MFC suspension show typical shear thinning behaviour in the interior part of the tube, in accordance with previous results obtained with conventional methods. The near wall behaviour shows existence of an apparent slip layer of thickness ~ 200 µm, most likely arising due to a concentration gradient created by hydrodynamical lift forces that induce migration of MFC particles away from the wall. In a sublayer of thickness ~ 100 µm, the fluid appears nearly Newtonian with viscosity value very close to that of pure water at the wall, and increasing with distance from the wall, along with concentration. Direct qualitative support for the existence of a low concentration region near the wall is obtained also by application of OCT in the imaging mode.

Lacking some fundamental drawbacks of conventional rheological techniques, the method based on flow velocity profiling by optical coherence tomography in the near-wall region, and by pulsed ultrasound velocimetry in the inner parts of the flow, appears a promising new technique for analysing the rheological and flow properties of many opaque complex fluids.

ACKNOWLEDGEMENTS

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REFERENCES


Transcription of Discussion

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Wolfgang Bauer \hspace{1cm} Graz University of Technology

I assume it was laminar flow that you were discussing in the paper; did you also try to go to turbulent flow?

Juha Salmela

Yes. As I mentioned, this part of the work is just about laminar flow because viscosity is well defined in laminar cases. We have also looked at turbulent conditions and that’s the work that we are currently very excited about, and we will publish it later. But this method can just as well be used for turbulent flows also but, as you know, viscosity is just not well defined there.

Lars Wågberg \hspace{1cm} KTH

Juha, thank you for a very nice presentation. With your high resolution measurements, I wonder if you are looking at single fibrils or are you looking at aggregates of fibrils?
**Discussion**

**Juha Salmela**

Good question. The best resolution of OCT (Optical Coherence Tomography) is a voxel size of $5 \, \mu m \times 5 \, \mu m \times 5 \, \mu m$ but, in our case, it is actually $5 \, \mu m \times 15 \, \mu m \times 15 \, \mu m$, so it is aggregates that we are looking at.

**Lars Wågberg**

If you did the measurements at pH 8, for instance, and at pH 2, would you see very different results?

**Juha Salmela**

I am not sure if that is already published in the previous work done with rheometers or if the publication is coming soon. It is known that there is a big dependency of flocculation on salt concentration and pH and similar things, so I would expect to see very different behaviour.

**Paul Krochak**  Innventia

I am wondering, because there is so much rheological data about MFC in all the different forms out there now and everybody kind of relies on it, is there any way to use the data that you are collecting and map that data back, or identify valid ranges? Can we interpret all the data that we have already based on your new measurements?

**Juha Salmela**

Excellent question, and, if you have this kind of flow between rheometer surfaces, I would say that it is not possible, and this is not even the worst case. I have to say that we also did many measurements using grooved surfaces, rough surfaces, and there was not a single case when we could see nice shearing. At this point, I would say it is very difficult to interpret rheometer data but we still have to do more work. All this work is still quite recent because we got the device just less than a year ago.

**Asaf Oko**  SP Technical Research Institution of Sweden

I thank you for a very nice presentation and images. Do you have a feeling that, if you can increase the separation between the water and the fibrillated cellulose, the solid content inside the pipe can be focused even more?
Very good idea. As I said, we have no idea about scaling yet, so I cannot say, but it will be very interesting when we go to smaller scales. I would assume that when you increase the pipe size, the lubrication layer does not scale up so, it may even remain at the 100 μm level. But when you approach the limit in pipe size, the topic becomes very interesting; there are some indications that we might be able to focus the fibres and orient them so that you can do some phase separation.

I have only one very simple question. It seems that you have qualitative behaviour which is far different from the classical behaviour of a suspension. Can you comment for example, on the phenomenon of “rollers”? Also, in the paper literature we can find some misunderstanding about whether we have a sub-laminar layer or not, can you comment on this please?

To me this behaviour actually is not far from previous models developed by Duffy et al.\(^1\) and recently modified by Jäsberg in his thesis\(^2\). The main difference is that we are saying that, at the wall, there is a viscosity profile rather than a thin layer of pure water and then the full concentration of pulp: there is actually a concentration profile and viscosity profile close to the wall. About the rollers: there are quite a few studies on these, and anyone who has a pipe line in which they can see the flow of a fibre suspension and who lets it start to flow very slowly, can actually see those rollers. So, qualitatively, the behaviour is quite similar to what was known.

But this is quite a coarse MFC grade and, if we go to grades like TEMPO oxidised MFC or similar grades that are transparent gel-like suspensions, the lubrication layer may be different, I do not know. These suspensions are somewhere between traditional fibre suspension and polymers.

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