Case Study

Carboxymethyl Cellulose (CMC) measurements in a 3” pipeline

The purpose of this document is to describe the intended use and operating principles of the OnLine Rheometer (OLR) built by Rheology Solutions Pty Ltd, and outline OLR performance during extended measurements of Carboxymethyl Cellulose (CMC) in our pilot-scale pipe-loop.

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1.1 TEST EQUIPMENT AND MATERIALS

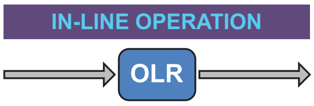
The OLR is a process rheometer, designed to continuously measure the flow properties of process liquids in-the-pipe. The intended use for the OLR is for process and/or quality control, whereby the plant operator and engineer may input specific quality or process control criteria with respect to the flow properties of their product, and monitor the conformance of the product in the pipe, as it passes through the OLR measuring cavity. The OLR was installed in an instrumented pipe-loop and data collected for CMC at various process flowrates.

1.2 OLR PRINCIPLE OF OPERATION

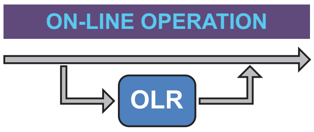
The OLR uses well established squeeze-flow kinematics. It measures storage and loss moduli by imposing a small cyclic deformation on a liquid sample at a variety of frequencies, from 1-100 Hz. Quality control parameters and user interface at plant operator level are based on η* (complex viscosity [Pa s]), as measured by the OLR.

As with a standard laboratory rheometer the response of the liquid is measured and displayed in terms of G’ (storage modulus [Pa]), G’’ (loss modulus [Pa]), δ (phase angle [Degrees]), and η*. These measurements are available at higher user levels in the OLR software, accessible to plant engineers and scientists, along with the possibility to add or alter QC settings for the plant operator.

The OLR can be installed in a process pipe in either of the two configurations shown in here. In the following we will refer to the configuration in which the OLR is directly placed in the pipe-line as the “in-line” mode of operation. When it is placed in a side-stream we will call it the “on-line” mode of operation. While operating in the on-line mode the OLR can be completely isolated from the main flow by the use of isolation valves and fluid can be intermittently routed through the side stream at convenient intervals for measurement purposes. Once the fluid is isolated the measurements occur in a geometry where the platens are completely submerged in the fluid of interest and there is no flow.



OLR in the process line



OLR in a side-stream

2. EXPERIMENTAL

Experiments in the in-line configuration were conducted in a pipe-loop built at our laboratory using a 2.5% solution of carboxymethyl cellulose (CMC) in water. The pipe-loop used consisted of a stainless steel tube of 70 millimetre internal diameter and rated to a maximum pressure of 10 bars.

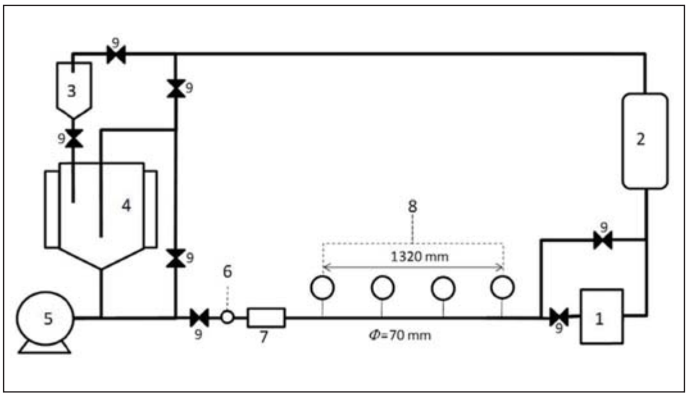


Figure 1. Schematic of the pipe-loop arrangement 1: OLR, 2: Flowmeter, 3: Measuring tank, 4: Bulk tank, 5: Mono pump, 6: Online Viscometer, 7: Inspection window, 8: Pressure transducers, 9: Valves

The loop was equipped with a flow meter that also allowed the measurement of single-point viscosity (as opposed to delivering a characteristic curve, unlike our OLR) and mass flow rate and the density of the fluid. The loop is also equipped with several temperature detectors (RTDs) and pressure gauges for monitoring the temperature and the pressure of the fluid during flow. A schematic diagram of the piping and instrumentation set-up is shown in Figure 1. As is shown in Figure 1 the OLR was configured directly in the flow path (in-line mode) in the experiments that follow. Experiments were conducted at various flow rates and the results were compared with measurements made using laboratory rheometer (Haake MARS III, Thermo Electron, USA) on samples drawn from the flow stream on each flow rate.

The pipe-loop shown in Figure 1 also allows the estimation of the viscosity using the measurement of the flow rate and the pressure drop. In experiments the fluid was pumped through the loop at various flow rates and the pressure drop over a known length of pipe-line was calculated. The pressure transducers were located at 10-30 pipe-diameters from the nearest bend to check for fully developed flow. The wall stress (τ_ω) was calculated as $\tau_\omega = D\Delta P/4L$ where D is the diameter of the pipe, ΔP is the pressure drop over the known section of the pipe, and L is the length of the pipe. The absolute value of the rate of shear at the wall ($\dot{\gamma}_\omega$) was calculated as $\dot{\gamma}_\omega, = (1+\frac{3n'}{4n'})\,{}^{8V}\!/_{D}$, where $V = 4Q/(\pi D^2)$ is the average velocity, Q being the volumetric flow rate. The factor n' was evaluated from experimental data using the following expression.

$$n^1 = \frac{\mathrm{d1n}\left({}^{D\Delta P}\!/_{4L}\right)}{\mathrm{d1n}\left({}^{8V}\!/_{D}\right)}$$

The factor n' defines the degree of non-Newtonian character in the fluid of interest.

The data from the OLR was independently verified using a state-of-the-art laboratory rheometer, a Thermo Scientific HAAKE MARS III.

3. RESULTS AND DISCUSSION

We start with a discussion of the results of the experiments performed with the OLR in the "in-line mode" in the pipe-loop introduced above. As mentioned previously we have use a 2.5% solution of CMC in water as our test fluid. The linear viscoelastic response of the fluid measured by Haake MARS III is shown in Figure 2.

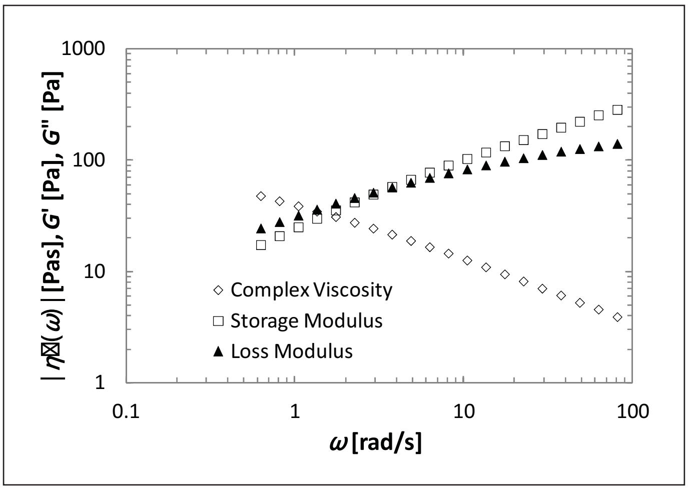


Figure 2. Dynamic modulii and the complex viscosity for 2.5% CMC solution in water. Crossover between the modulii occurs at about ω≅4 rad/s.

In Figure 2 the storage modulus (G'), the loss modulus (G") and the modulus of the complex viscosity ($|\eta^*|$) are plotted against the angular frequency (ω). The frequency range was chosen to allow for appropriate overlap between the measurements made on the laboratory rheometer and the OLR. From Figure 2 an estimate of the relaxation time of the fluid can be obtained as $\lambda = 1/\omega^*$, where ω^* is the angular frequency at which the storage and the loss moduli cross. For the fluid under consideration $\lambda \approx 0.2$ seconds at 25°C.

An important parameter in pipe-flow experiments is the pressure drop (ΔP) at the wall measured over a known distance in a pipe. Pressure drop data is generally useful for the estimation of viscosity when the flow conditions are laminar and elastic effects invoked by flow are unimportant.

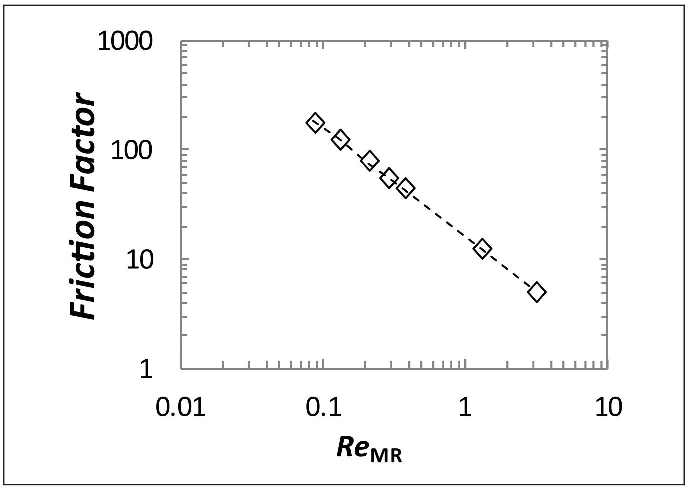


Figure 3. Plot of friction factor and the Metzner-Reed Reynolds Number for the experiments conducted (symbol). The line in the figure is 16/ReMR. The figure shows that the experiments were conducted in laminar flow conditions and is a convincing argument for the accuracy of pipe-loop measurements.

In Figure 3 we plot the friction factor (-) against the Reynolds number (Re_{MR}). It can be observed from Figure 3that the data is well represented by the $16/Re_{MR}$ line suggesting that the flow remained laminar in these experiments. The close agreement between the measured friction factor data with the line $16/Re_{MR}$ also illustrates that the instrumentation in the pipe-loop is functioning well and delivering reliable data.

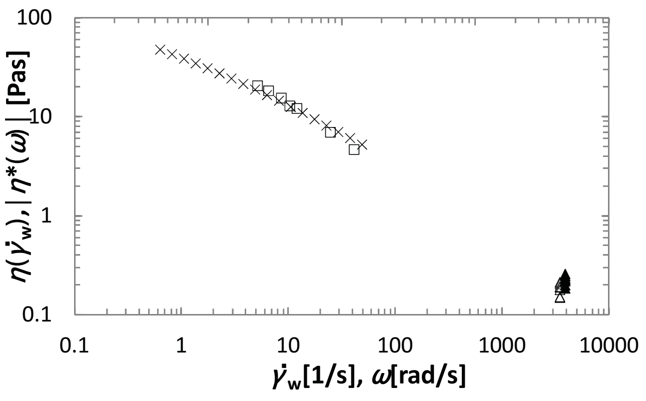
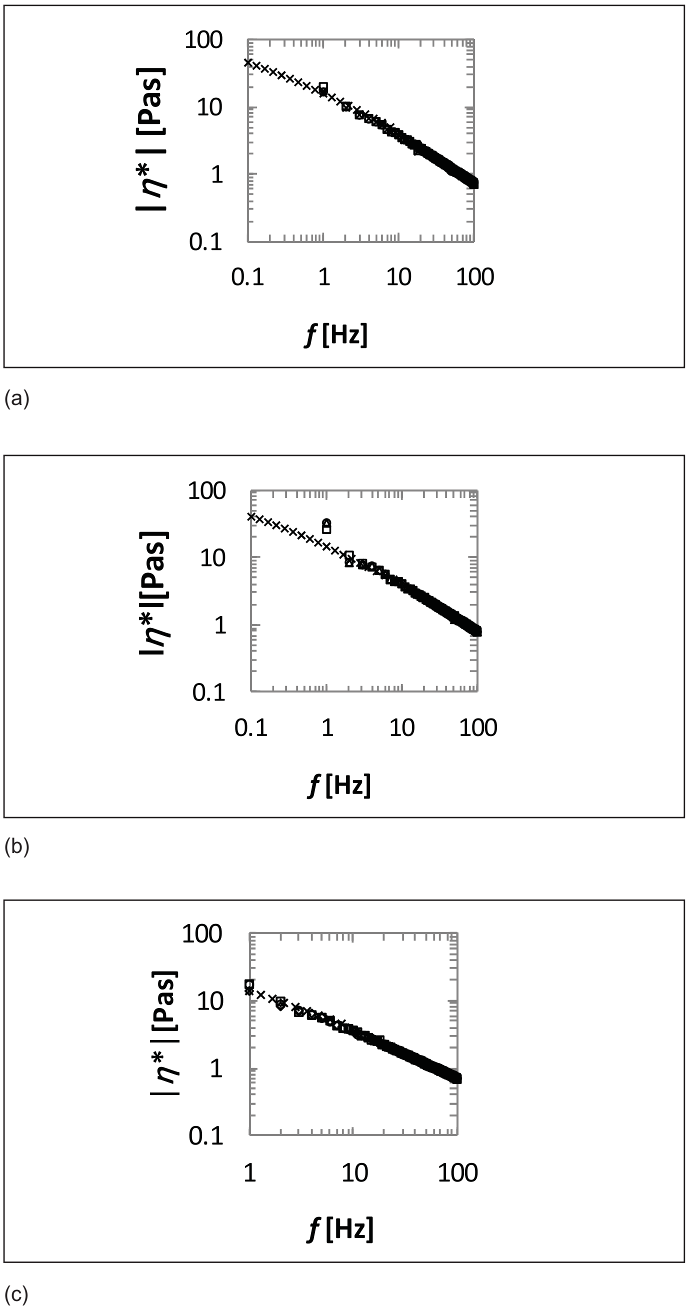


Figure 4. Viscosity measured using pressure drop in a flowing pipe (□), and using popular process viscometers (△) & (▲), compared with the complex viscosity measured in the laboratory (x).

In Figure 4 we present the results of the pipe-flow experiments in a plot of viscosity $\eta(\dot{\gamma}_\omega) = \tau_\omega / \dot{\gamma}_\omega$ against $\dot{\gamma}_\omega$ using unfilled squares as makers.

The viscosity is observed to decrease with increasing shear rate. Rheological data obtained in steady shear flows can be compared to those obtained in small-amplitude oscillatory-shear experiments when the empirical Cox-Merz rule holds. Cox-Merz rule suggests that the magnitude of the complex viscosity in polymeric fluids can be compared with the viscosity at equal values of angular frequency and shear-rate. We therefore compare the results with data on the complex viscosity, $|\eta^*|$ obtained from small-amplitude-oscillatory-shear experiments (crosses) with the viscometric data available from the pipe flow experiments. As can be observed from Figure 4 the correspondence is reasonably favourable although two data-points at the highest shear-rates appear to diverge slightly from the trend demonstrated by the complex viscosity. This could well be a consequence of the transient nature of the flow at high Reynolds numbers.

Also shown in Figure 4 are the viscosities measured by the two commercially available on- line viscometers. It can be observed that the viscometers operate at strikingly higher values of shear-rate and measure a viscosity that is at least an order of magnitude lower than those measured by the laboratory rheometers and the pipe-flow experiments. There is also a noticeable scatter in the values of the measured viscosity.



(a)

(b)

(c)

Figure 5. Results of experiments conducted at various flow-rates. The measurements of the laboratory rheometer are represented using cross symbols (X). The other symbols represent measurements made by the OLR for repeated experiments at a fixed flow-rate. Results from three representative flow rates are shown: (a) 1500 kgs/hr (≈0.11m/s) , (b) 1900 kgs/hr (≈0.14m/s) and (c) 2900kgs/hr (≈0.21 m/s).

In Figure 5 we present the data measured by the OLR that operated as a part of the pipe-flow experiments for three separate experiments and compare them with the complex viscosity data presented before in Figure 4. It can be observed that the measurements from the OLR agree well with the measurements performed in a specialised laboratory rheometer. As mentioned previously the OLR was placed “in-line” with flow in these experiments. It is envisaged that in actual practice any problem at high flow rates can be easily circumvented by placing the OLR in a side-stream equipped with isolation valves and adopting a strategy where the measurements are conducted intermittently by diverting the process flow through the side-stream.

4. SUMMARY AND CONCLUSIONS

The OLR used in-line (liquid flowing through the measurement chamber) has been shown to give results which agree well with a laboratory rheometer. The test material (CMC) has been well characterised by the OLR and the experimental apparatus used has given independent confirmation that the measurements of the OLR are reliable and representative.

The Rheology Solutions pilot plant has proven an effective test site for CMC, and the instrumentation has been found to provide accurate and useful data.
Future work should involve using the pilot plant to evaluate other materials of interest and as a consequence the capacity and capabilities of the pipe-loop are outlined here:

RHEOLOGY SOLUTIONS PIPE LOOP PILOT-PLANT		
Bulk Tank	Materials	316 SS
	Volumetric capacity	350-500 l
	Mixing/Agitation	2 x twin-, pitched-blade, 60rpm
	Baffles	None
Pump	Mono pump	5kW, 200rpm, 20m^3/hr (water)
Temperature Control	Ambient – 70°C	Band heater on bulk tank.
		Heat tracing on pipework.
Pipework	Materials	316 SS Tube
	Diameter	3" Tube
	Connections	3" Tri-Clover
	Pressure rating	0-10 bar g
Instrumentation	Temperature	3-wire RTDs (bulk tank and pipeline)
	Pressure (0-20 bar g)	Ceramic diaphragm
	Mass flow meter	Coriolis type
	In-line viscometer	Single-point measurement types
	OnLine Rheometer	In-line or on-line configuration

Enquiries can be made for pilot plant testing by contacting Rheology Solutions OnLine Rheometer Group or by completing the Pipe Loop Enquiry Form.

the **OLR** *keeps your process in line*

Rheology Solutions

The **OnLine Rheometer Group** is a division of **Rheology Solutions**
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