Technical White Paper

High speed, small sample rheometric screening using the Cambridge viscometer

High speed, small sample rheometric screening using the Cambridge Viscosity viscometer

A. Summary

Characterizing a fluid's rheology rapidly while using only a small amount of fluid (<2mL) is highly advantageous in many applications. Unfortunately, this has not historically been possible in most instances. In fact, such characterizations require significant time, fluid, and technician skill.

Cambridge Viscosity has demonstrated that accurate, rapid, rheometric characterization with a small amount of fluid is now possible with their unique electromagnetic viscometer technology. As reported in this paper, detailed characterizations of Newtonian and non-Newtonian fluids were performed with a Bohlin rheometer; then these fluids were analyzed using the Cambridge device. In each case, the results from the Cambridge data correctly identified the different fluids by their rheometrical structures. Specific findings are summarized below.

1. The Cambridge viscometer shows excellent agreement with the Bohlin rheometer with fluids throughout the 50-5000mPas range.
    a. Newtonian and Non-Newtonian fluid systems were successfully identified using relatively few data points; Cambridge's results comparing favourably with the Bohlin values.
    b. Measurement time for non-Newtonian samples with a single Cambridge sensor in these preliminary tests was 7 minutes (primarily due to the recovery time after shear down). This time would be much shorter for Newtonian liquids where the recovery time is essentially zero.
2. The Cambridge unit is reliable and easy to use.
    a. The Cambridge unit was operated to control shear history for data uniformity.
    b. The range of shear rates achievable with any particular piston in the Cambridge device is limited, so a number of pistons with overlapping ranges may be required, depending on the fluid characteristics.
    c. The sensor is easy to clean mechanically, and lends itself to automatic, hands-off cleaning.

B. Newtonian Fluid Systems

Two fluids, "N1" and "N2", are Newtonian liquids with viscosities measured on the Bohlin CVOHR rheometer of 235mPas and 65mPas respectively at 20°C. Results with the Cambridge viscometer agreed well with the Bohlin.

In the initial test set-up of the Cambridge device, a water bath was simply maintained at a constant temperature of 20°C with no direct temperature control between the sensor and the bath. In addition, the following protocol was used (referred to as protocol 1).

1. Place sample in cell
2. Insert piston
3. Leave to rest for 5 minutes
4. Measure for 60s at low shear
5. Measure at high shear for about 60-100s.

The data shown below is obtained for the N1 (Figure 1) and N2 (Figure 2) fluids with the Cambridge Viscometer. In each case, the change in shear rate from low to high occurred around the 100s mark. Each fluid is accurately shown as Newtonian.

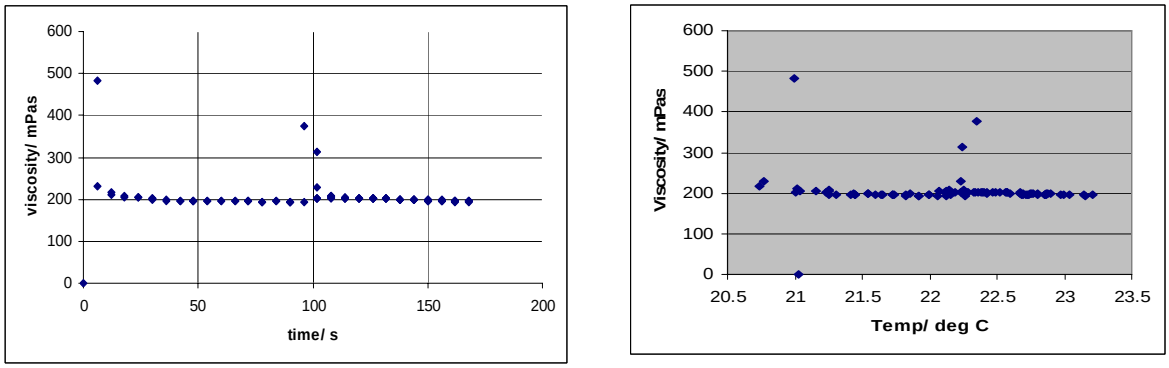

Fig 1: Viscosity of N1 as a function of time and temperature, 50-1000 range, as measured with the Cambridge viscometer using protocol 1

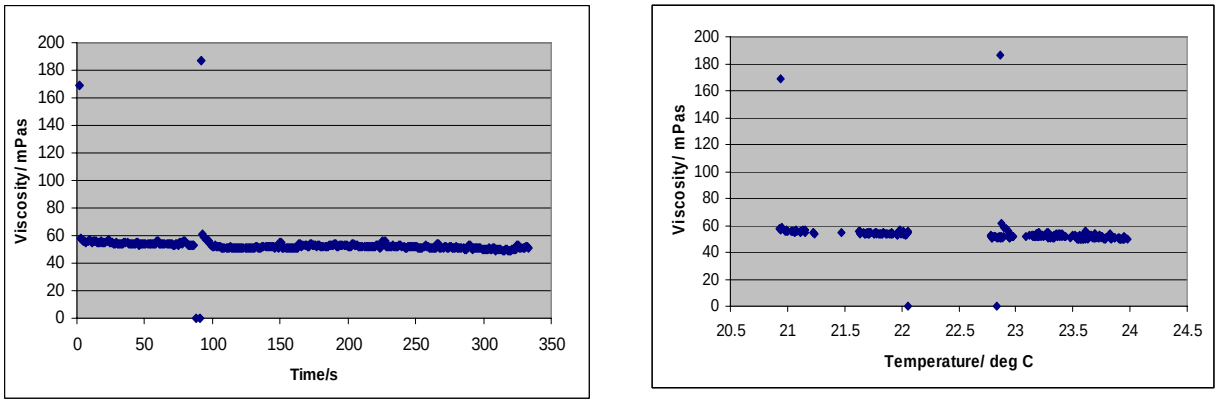

Fig 2: Viscosity of N2 as a function of time temperature, 50-1000range, as measured with the Cambridge viscometer using protocol 1

B) Non-Newtonian Fluid Systems

A range of non-Newtonian systems were measured on the Cambridge viscometer using different shear rates to develop a shear profile for a screening level of rheological characterization and the results compared to the detailed rheological data obtained on that fluid with Bohlin CVO rheometer.

The tests were designed to simulate a rapid throughput rheological analysis: the sample was placed in the Cambridge viscometer then the piston was introduced into the sample and then powered. By doing this, the sample was subjected first to a high shear, which functioned as a 'pre-shear' to remove rheological history from the sample; then the sample was left to equilibrate for 5 minutes to recover and then viscosity measurements were made at the two current settings. Where applicable, this was repeated with a second (and sometimes a third) piston to generate data at additional shear rates.

Measurements with the Bohlin instrument followed a similar regimen. The sample was first subjected to a high shear to remove the rheological history and then allowed to equilibrate. Then a series of discrete series of shear rates (0-600s^{-1}) were used with the Bohlin to determine the shear rate shear stress profile of the fluid. All measurements were carried out with the temperature bath set to 20°C.

Initial Non-Newtonian Fluid Tests

Initially an aqueous suspension of a bentonite clay that swells was used. In general, good agreement was obtained as far as the samples would allow (due to some ageing effects in the suspensions). The Cambridge system identified these samples as being non-Newtonian and found Casson viscosities similar to those obtained on the Bohlin.

Next, a series of suspension concentrate formulations were measured. These were prepared and stored so that they would show little in the way of further aging effects. The samples chosen had a range of Casson viscosities from about 70mPas to 300mPas (from the Bohlin). The results obtained on both instruments are shown in Table 1 and Figures 3 - 5 below.

	Bohlin		Cambridge	
SC#	η_c/ mPas	σ_c/ Pa	η_c/ mPas	σ_c/ Pa
1	154	1.78	137	1.67
2	70.7	1.98	55.7	2.38
3	157	2.23	152	1.62
4	303	9.85,6.38	430	2.51

Table 1: Casson yield stress (σ_c) and viscosity ($\eta_{c)}$) for the 4 suspension concentrations (SC)

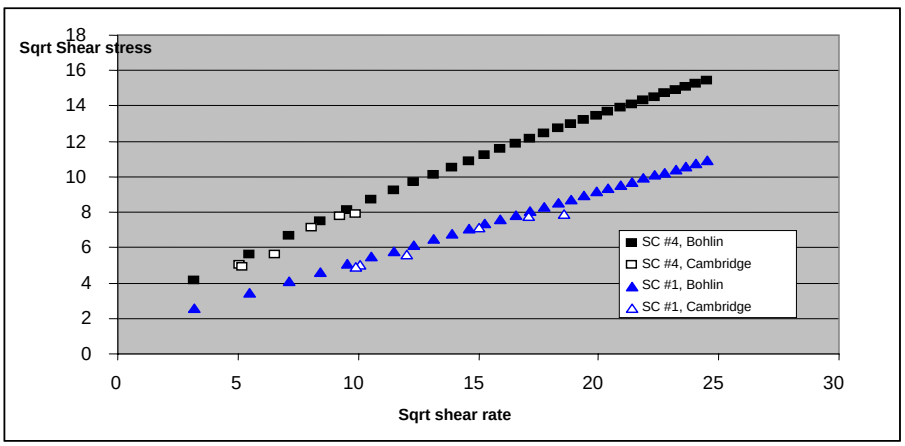

Figure 3: Casson plots for SCs 1 and 4 obtained from the two instruments

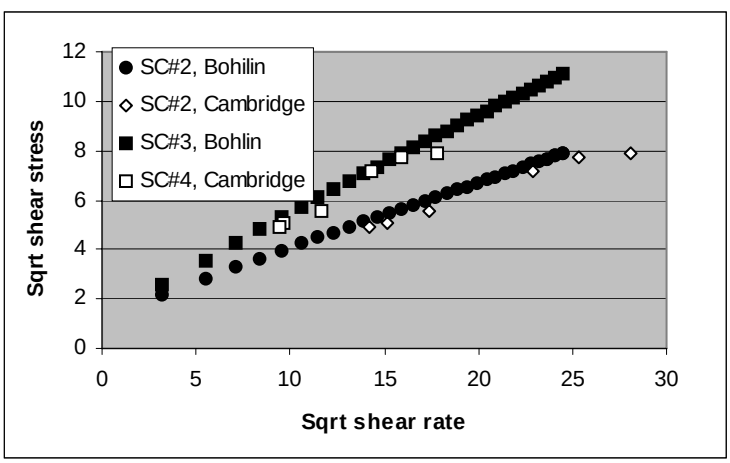

Fig. 4: Casson plots for SCs 2 and 3 obtained from the two instruments

The data from the Cambridge system was obtained with three of the pistons. In general, two of the pistons actually fully covered the viscosity range of the samples, while the third was slightly outside the calibrated range (at least on one point). This is evident in figs. 3 and 4, where the point of the highest shear rate for each of the suspensions dipped down below the extrapolated line from the lower shear rates on the Casson plots of $\sigma_c^{1/2}$ against $\eta_c^{1/2}$. There are minor differences in the values for the yield stress and viscosity between the two instruments for suspensions 1-4, but are acceptable in terms of a high throughput situation. These differences, especially those in the yield stress, are at least partly attributable to the differences in shear history.

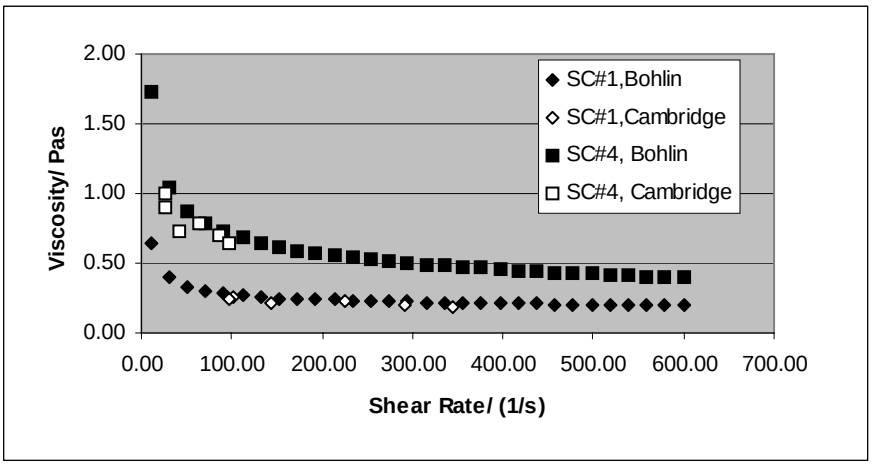

Fig. 5: Viscosity versus shear rate plots for SCs 1 and 4

The shear-down in the Bohlin was done at 300s^{-1} whereas the actual shear rate in the Cambridge as the piston was put into the sample was unknown and different for each sample, since it was put in by hand. It would certainly have been much greater than 1000s^{-1} for the lower viscosity samples. The greatest discrepancy was with the most viscous sample, SC#4, where the increased viscosity would tend to increase the time required for any structure to fully reform after shear-down. Moreover, the shear rates attainable in the Cambridge viscometer were very much at the low end of the shear rate region (25-100s^{-1}). Analysis of such a small range will necessarily introduce larger errors into the analysis, hence the greater discrepancy in this region.

Additional Non-Newtonian Fluid Tests

In another set of experiments, three suspension concentrates (SC#5-7) of similar properties were tested on both instruments, and again good agreement (within 10%) was obtained. Greater differences were seen in the yield stresses, but again that was likely to be due to differences in shear history and small number of data points from the Cambridge.

	Bohlin		Cambridge	
SC#	η_c/ mPas	σ_c/ Pa	η_c/ mPas	σ_c/ Pa
5	30.4	4.59	27.3	6.17
4	26.5	5.0	24.7	6.50
6	24.6	5.63	24.9	6.16

Table 2: Casson viscosities and yield stresses for suspension 5-7

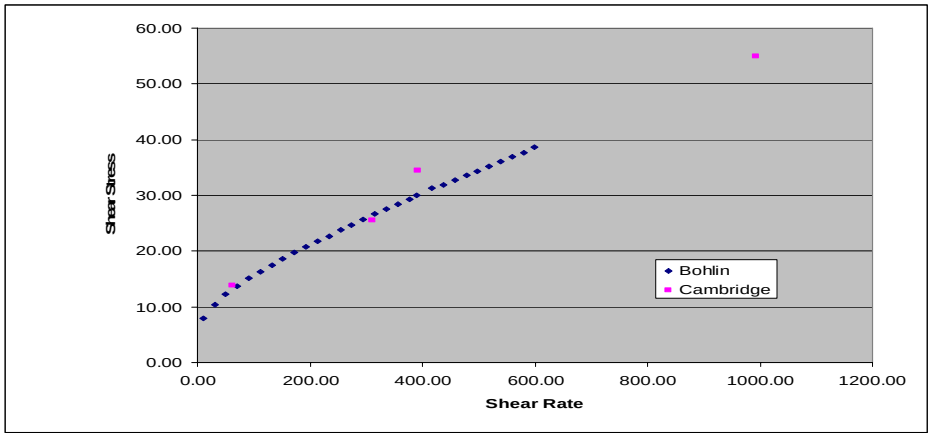

Fig 6: Shear rate shear stress plot for SC#5

Two commercial lattices were then measured. These were 'A', a vinyl acetate-vinyl chloride-ethylene- acrylic copolymer and 'B', a vinyl- acetate-ethylene copolymer. These are quite difficult to handle samples, similar to PVA glue. These were both non-Newtonian and their shear stress shear rate curves are shown below.

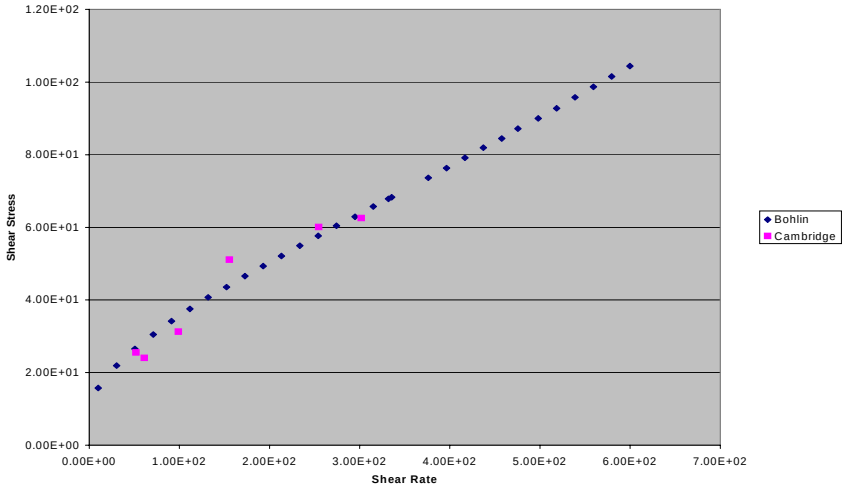

Fig. 7: Shear stress shear rate plot for latex A.

The agreement between the Bohlin and Cambridge instruments for latex A is very good, and the shear rates available with the set of pistons available for these experiments covered a reasonable range (up to around $300s^{-1}$). On the other hand, the shear rates with the more viscous latex B (high shear viscosity ca. 1500mPas compared to 200mPas for latex A) were much more limited, up to only ca. $60s^{-1}$. This does highlight the fact that a) a large range of pistons will be required to be able to cover a useful range of shear rates and b) some degree of intelligence will be required in the system to choose a suitable piston.

A concentrated non-aqueous polystyrene solution was also tested. In this case, the Cambridge gave a viscosity (250-5000mPas piston) of 4.9Pas, however on the Bohlin the sample was seen to undergo shear fracture where the viscosity underwent a discontinuous fall from 7Pas down to 2Pas at a shear rate of $400s^{-1}$. Clearly, the Cambridge was giving the correct order of magnitude for the viscosity, being in between the two extremes found on the Bohlin, but little more can be drawn from this experiment due to the complex nature of the rheology.

Other non-Newtonian polymer solutions have been tested and shown good agreement. And the poorer agreement with the non-aqueous PS solution was due to the unexpected shear fracturing. One commercial product measured showed poor agreement between the two instruments, and the reason why this was so is be considered.

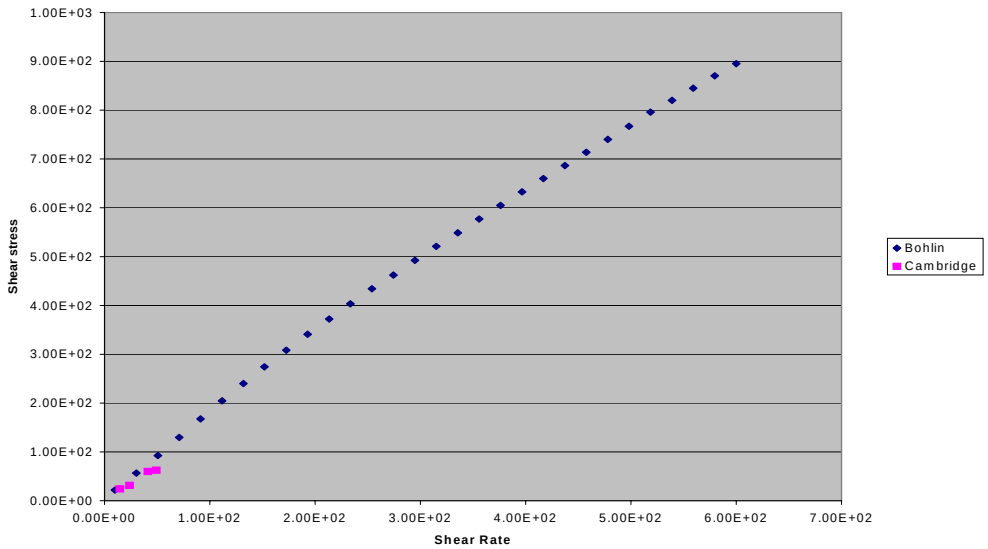

Fig 8: Shear stress shear rate plot for latex B.

Conclusions

The Cambridge viscometer has been evaluated as a high throughput rheometric screening device. This evaluation involved testing the device in a variety of Newtonian and non-Newtonian fluid systems ranging from concentrated suspensions to highly viscous polymer solutions. In all cases, Cambridge measurements compared well with a Bohlin rheometer. The results indicate the Cambridge viscometer should be considered anywhere rapid rheometric screening with a small sample of fluid (<2mL) is required.

For more information contact: