APPLICATIONS OF CAPILLARY VISCOMETRY IN CANE SUGAR FACTORIES

LIONNET G R E and PILLAY M

Tongaat-Hulett Sugar Ltd, Private Bag 3, Glenashley, 4022, South Africa
raoul.lionnet@huletts.co.za   melanie.pillay@huletts.co.za

Abstract

The importance of viscosity in cane sugar processing is well established, particularly as far as exhaustion is concerned. Techniques for the determination of the viscosity and consistency of massecuites and of molasses have therefore been the subject of many investigations. Two types of instruments, the Brookfield rotating cylinder viscometer and the pipeline viscometer, have been utilised, with the former being used more extensively. Another type of instrument, the gravity driven glass capillary viscometer, is used by national standard organisations; it yields absolute viscosity data of high accuracy and with good precision.

This paper describes a number of applications for glass capillary viscometry. The technique was used to determine the concentrations of the usual clarification flocculant in water and in juices; to compare the viscosities of limed juice, clear juice and of diluted syrup; to measure the viscosity of A-molasses from different factories, and to carry out preliminary work on the effect of suspended solids on the viscosity of final molasses. The viscometers are inexpensive and easy to use.

Finally, double tube capillary viscometers are briefly mentioned.

Keywords: capillary, viscosity, viscometry, flow behaviour, flocculant, molasses, factory process

Introduction

The importance of viscosity in cane sugar processing is well established and the literature contains many publications on this topic. Much of the work that is relevant here has been done in Australia (Ness, 1980, 1984; Broadfoot and Miller, 1990; Broadfoot *et al*, 1998) and in South Africa (Rouillard and Koenig, 1980; Durgueil, 1987; Barker, 1998); in addition, the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) describes the procedures for measuring the apparent viscosity of molasses, which were officially adopted in 1994 (Anon, 1994).

There are a number of different viscometric techniques for measuring the flow behaviour of fluids; the literature mentioned above shows that the rotating cylinder method has been widely used in the sugar industry, and that the Brookfield viscometer is one make of rotational viscometer that is commonly used for work with molasses and massecuites; it is, however, less suitable for low Brix materials such as juices. Pipeline viscometers and glass capillary viscometers are mentioned in the Australian literature, but do not seem to have been used in South Africa.

Residual concentrations of the usual clarification flocculants in juice and in sugar can have negative effects. In factories returning clarifier mud to the diffuser, any excess flocculant present in the mud could reduce percolation in diffusers, thus promoting flooding. Attempts

to quantify residual concentrations of flocculants in juices by conventional chemical analysis have not been successful, mostly because it has not been possible to find a simple analysis yielding a result that could be related clearly to the concentration of flocculant. Capillary viscometry was investigated in an attempt to solve this problem and to see whether the technique could be useful elsewhere in cane sugar processing.

Theory

When a liquid flows through a capillary of a material which it wets, emerging with a small velocity, the volume v which passes a section of the tube in a time t is given by Poiseuille's equation:

$$v = \frac{\pi p t r^4}{8 l \eta}$$

where p is the pressure difference between the ends of the tube of length l and radius r; η is the dynamic viscosity expressed in units of N.s.m^{-2} or Pa.s. It is usually sufficient to compare the viscosity of the liquid being investigated with that of a standard (water or a sucrose solution of known concentration), by measuring the times taken for equal volumes of the two liquids to flow through the same capillary under pressures due to their own weights. The densities of both liquids must be known; the viscosity of the liquid can then be obtained knowing the viscosity of the standard (James and Prichard, 1974). The terms 'viscosity' and 'dynamic viscosity' are used interchangeably in the paper and refer to the viscosity defined above.

A typical gravity driven glass capillary viscometer such as the Ostwald or U-tube viscometer can be used. If η_1 and η_2 are the viscosities of the two liquids of densities ρ_1 and ρ_2, while t_1 and t_2 are the times of flow, it follows from Poiseuille's equation that:

$$\frac{\eta_1}{\eta_2} = \frac{t_1 \rho_1}{t_2 \rho_2}$$

Thus, if the viscosity of the standard is known, then that of the given liquid can be determined. Temperature must obviously be known and controlled.

The velocity of flow of a liquid through a capillary is proportional to the fourth power of the radius of the tube. To cover a wide range of viscosities, it is necessary to have a number of viscometers with tubes of different radii.

Results

The viscosity of flocculant solutions

The Sugar Milling Research Institute was requested to carry out an exploratory investigation. Pure sucrose solutions (10.0 Brix) containing known concentrations of a given clarification flocculant were tested against water, at 20ºC in a Poulten Selfe and Lee Ubbelohde-type PSL ASTM-IP 1C capillary viscometer. The results (personal communication[1]) are in Table 1. Means and standard deviations are included; the precision, based on coefficients of variation (CV), is considered excellent.

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[1] Steve Davis, Sugar Milling Research Institute, University of KwaZulu-Natal Durban, South Africa.

Table 1. Flow times in a capillary viscometer for water and 10 Brix sucrose solutions at 20ºC containing known concentrations of flocculant.

Replicate	Flow time (s)				
	Water	Flocculant conc. (mg/l) in 10 Brix sucrose solutions			
		0	5	10	20
1	22.46	28.07	28.71	29.33	30.43
2	22.43	28.10	28.69	29.39	30.37
3	22.48	28.11	28.69	29.29	30.47
4	22.45	28.12	28.53	29.35	30.47
5	22.49	28.13	28.76	29.18	30.33
6	22.48	28.20	28.67	29.25	30.47
7	22.47	28.13	28.66	29.22	30.32
8	22.45	28.02	28.61	29.22	30.49
9	22.46	28.13	28.69	29.20	30.40
10	22.53	28.13	28.62	29.24	30.33
Mean	22.47	28.11	28.66	29.27	30.41
Std dev	0.027	0.046	0.063	0.070	0.067
CV	0.1	0.2	0.2	0.2	0.2

These results are now used to calculate the dynamic viscosities. The viscosity and density of water at 20ºC are 1.00×10^{-3} Pa.s and 998.20 kg.m^{-3} respectively; for 10.0 Brix sucrose solutions the corresponding values are 1.29×10^{-3} Pa.s and 1038.12 kg.m^{-3} (Bubnik *et al*, 1995). The calculated viscosities are in Table 2.

Table 2. Viscosities of 10 Brix sucrose solutions at 20ºC containing knownconcentrations of flocculant.

Parameter	Water	Flocculant conc. (mg/l) in 10 Brix sucrose solutions			
		0	5	10	20
Time (s)	22.47	28.11	28.66	29.27	30.41
Viscosity (Pa.s)	1.00×10^{-3}	1.30×10^{-3}	1.33×10^{-3}	1.35×10^{-3}	1.41×10^{-3}

The value of 1.30×10^{-3} Pa.s for the 10 Brix sucrose solutions without any flocculant agrees well with the value given by Bubnik *et al* (1995) (1.29×10^{-3} Pa.s), showing that the technique is also accurate. The viscosities were plotted against flocculant concentrations in Figure 1.
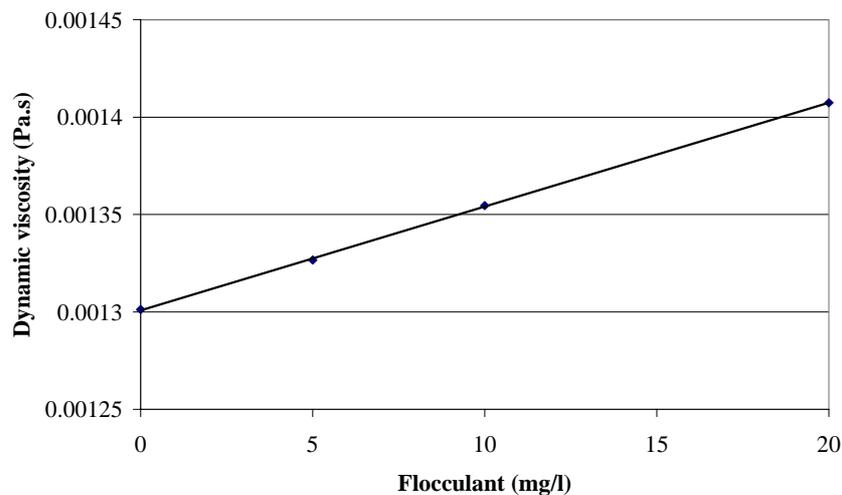
Figure 1. The effect of flocculant on the viscosity of 10 Brix sucrose solutions at 20ºC.

A linear regression yields Equation 1:

$$\text{Viscosity} = 5.33 \times 10^{-6} \times \text{conc. of flocculant} + 0.00130 \quad \ldots\ldots\ldots\ldots(1)$$

where the viscosity is in Pa.s and the concentration in mg flocculant per litre of solution. The R^2 value was 0.9998 for four pairs of observations; clearly, the relation is strongly linear at these concentrations.

These results indicate that capillary viscometry can provide a precise and accurate approach to investigate the concentration of flocculant in sucrose solutions.

Under industrial conditions an operator may need to check the concentration of the flocculant stock solution that has been prepared in the plant. Capillary viscometry could be used for this check, by measuring the viscosity of flocculant solutions of known concentrations to produce a calibration against which the viscosity of the plant solution would be compared. This approach was tested at a factory; the results are in Table 3 and Figure 2; the densities of the flocculant solutions were assumed to be the same as that of water.

Table 3. Viscosities of industrial flocculant solutions at 20ºC.

Parameter	Water	Conc. of standard flocculant solutions (% m/m)					Plant solution
		0.10	0.05	0.025	0.0125	0.00625	
Time (s)	10.41	71.66	33.30	18.36	13.11	11.93	50.73
Visc. (Pa.s)	1.00×10^{-3}	6.88×10^{-3}	3.20×10^{-3}	1.76×10^{-3}	1.26×10^{-3}	1.15×10^{-3}	4.87×10^{-3}
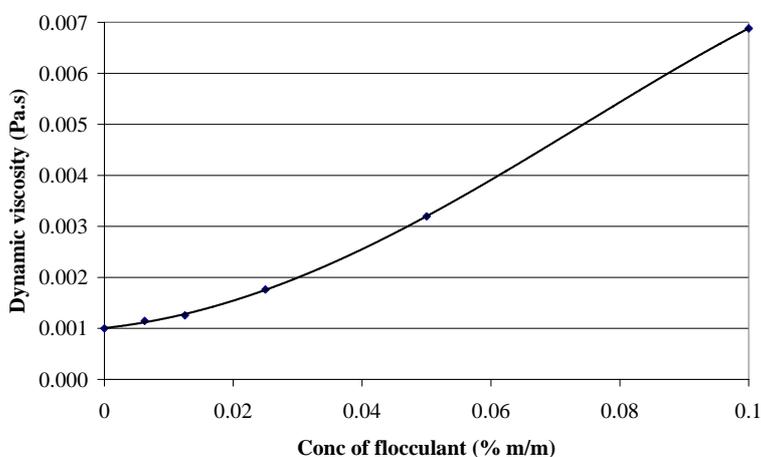
Figure 2. The effect of concentration on the capillary viscosity of flocculant solutions at 20ºC.

The relation, over the wider range of concentrations, is now non-linear; Equation 2 was obtained:

$$\text{Viscosity} = 0.001 \times e^{19.7 \times \text{conc of polyelectrolyte}} \quad \ldots\ldots\ldots\ldots..(2)$$

where the concentration is on a mass flocculant % mass solution basis, and the viscosity is in Pa.s. The regression gave an R^2 value of 0.988 for six pairs of observations. The concentration of flocculant in the plant stock solution can be calculated to be 0.08%; this result may now be compared to the concentration decided upon for optimum performance in

the plant. This approach was not tested exhaustively across the range of flocculants used in the industry, but the results obtained above are certainly encouraging.

The presence of higher than normal concentrations of flocculant in mud could impact negatively on diffuser operations when mud is routed to the diffuser. The use of capillary viscometry to monitor the viscosities of limed juice, clear juice and of the juice in mud, from different factories (M1, M2 and M3), was investigated by measuring the viscosity of catch samples diluted to 10 Brix. Results are in Table 4; there is no evidence of higher viscosities in the juice from mud. Generally, all the juices show viscosities between 1.3×10^{-3} and 1.4×10^{-3} Pa.s; this compares to a value of 1.29×10^{-3} Pa.s for a 10 Brix pure sucrose solution, indicating that the viscosity of the juices is only about 5% higher than that of a pure sucrose solution.

**Table 4. Viscosity (Pa.s) at 20ºC of juices diluted to 10 Brix
(Standard: 10 Brix sucrose solution).**

Factories	Limed juice	Clear juice	Juice in mud
M1	1.39×10^{-3}	1.34×10^{-3}	1.36×10^{-3}
	1.41×10^{-3}	1.35×10^{-3}	1.37×10^{-3}
	1.39×10^{-3}	1.35×10^{-3}	1.36×10^{-3}
M2	1.31×10^{-3}	1.29×10^{-3}	1.30×10^{-3}
M3	1.35×10^{-3}	1.30×10^{-3}	1.39×10^{-3}

The viscosity of juices and of syrup

Weekly composite samples of limed juice, clear juice, mud and evaporator syrup were taken at a factory (M1) and used to measure viscosities at 20ºC and 10 Brix, using a 10 Brix sucrose solution as the standard. The results are in Table 5.

Table 5. Viscosity (Pa.s) at 20ºC of weekly composites at M1, diluted to 10 Brix.

Week	Limed juice	Clear juice	Juice in mud	Syrup
03/11 to 10/11	1.30×10^{-3}	1.28×10^{-3}	1.30×10^{-3}	1.41×10^{-3}
11/11 to 17/11	1.37×10^{-3}	1.32×10^{-3}	1.39×10^{-3}	1.44×10^{-3}
18/11 to 24/11	1.40×10^{-3}	1.35×10^{-3}	1.41×10^{-3}	1.48×10^{-3}

The viscosities (at 10 Brix) of limed juice, of clear juice and of juice from mud are similar, but that of syrup is about 10% higher, indicating that the evaporation process could increase viscosity. It is also evident that viscosities increased over the month of November at M1.

The viscosity of A-molasses

During recent A-centrifugal tests at one of the Tongaat-Hulett factories, it became necessary to investigate the viscosity of the A-molasses. Samples of A-molasses were obtained from a number of factories; the samples were diluted over a range of 40 to 60 Brix and the viscosities determined in a glass capillary viscometer using a refined sugar solution of 50 Brix as the standard; all the determinations were done at 20ºC. The results are shown in Figure 3, where the letter M identifies the factories; exponential equations fitted the data well and the equations were used to estimate the viscosity at 70 Brix. M1, where the centrifugal investigation was done, clearly shows higher viscosities.
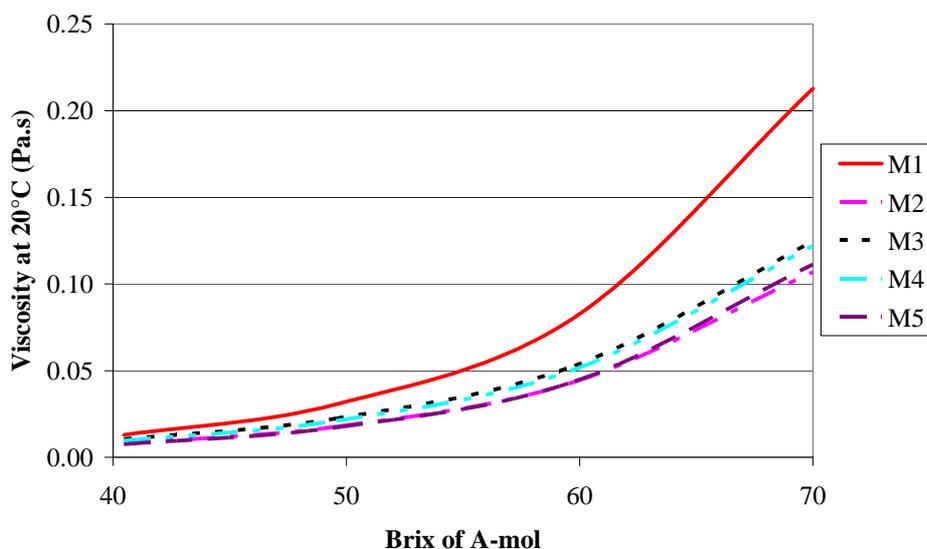
Figure 3. The viscosity of A-molasses from different factories.

The viscosity of final molasses

The results obtained here are very preliminary and have been included only to highlight another possible application of capillary viscometry. Final molasses was diluted to 65 Brix; part of the diluted sample was kept as is, while the rest was centrifuged at 2500 G-force for five minutes. Viscosities were determined at 20ºC on the as is and on the centrifuged molasses over a range of Brix values from 65 to 40. Exponential equations were then fitted and viscosities estimated at 60, 70 and 80 Brix. The test was done with molasses from two factories (M1 and M2); the results are in Table 6. Although the centrifugation process removed some suspended matter (assessed visually), this appears to have had no measurable effect on the viscosity. As expected, final molasses shows higher viscosities (30 to 70%) than a refined sugar solution at the same Brix; finally, the molasses from factory M2 shows viscosity values of between 25 and 30% higher than those from factory M1.

Table 6. The viscosity of 'as is' and centrifuged final molasses.

Brix	M1		M2		Refined sugar solution (std)
	Viscosity at 20ºC (Pa.s)				
	As is	Centrifuged	As is	Centrifuged	–
60	0.075	0.077	0.095	0.097	0.058
70	0.24	0.25	0.32	0.32	–
80	0.78	0.81	1.05	1.07	–

Double tube capillary viscometers

At this stage only liquids have been investigated here; Bruhns (2004) describes a double tube viscometer in which the flow properties of massecuites can be investigated. The suspension flows simultaneously through tubes of different diameters but with the same length/diameter ratio, thus giving the same shear stress in both tubes. The technique also yields relative viscosities for the suspensions.

Conclusions

This work deals only with practical applications of capillary viscometry in cane sugar factories.

Bearing the above limitation in mind, capillary viscometry has been shown to be both precise and accurate for the measurement and comparison of viscosity in juices and syrups diluted to a Brix of 10. It is also suitable for the determination of the concentration of flocculant in factory streams. Furthermore, it can be used to compare the viscosities of molasses, after dilution to Brix values around 60.

The equipment is inexpensive and its use involves simple procedures that could easily be carried out at factories. It is particularly useful for comparing the viscosities of different samples.

REFERENCES

Anon (1994). ICUMSA Methods Book with first (1994) and second (2000) supplements. SPS-5: 1-10.

Barker B (1998). Theoretical and practical considerations on the rheology of sugar products. *Proc S Afr Sug Technol Ass* 72: 300-305.

Broadfoot R and Miller KF (1990). Rheological studies of massecuites and molasses. *Int Sug J* 92: 107-146.

Broadfoot R, Miller KF and McLaughlin RL (1998). Rheology of high grade massecuites. *Proc Aust Soc Sug Cane Technol* 20: 388-397.

Bruhns M (2004). The viscosity of massecuite and its suitability for centrifuging. *Zuckerindustrie* 129(12): 853-863.

Bubnik Z, Kadlec P, Urban D and Bruhns M (1995). Sugar Technologists' Manual. 8th Edition, Bartens.

Durgueil EJ (1987). Determination of the consistency of non-Newtonian fluids using a Brookfeild HBT viscometer. *Proc S Afr Sug Technol Ass* 61: 32-39.

James AM and Prichard FE (1974). *Practical Physical Chemistry*. 3rd Edition, Longman, UK.

Ness JN (1980). Massecuite viscosity - some observations with pipeline viscometers. *Proc Aust Soc Sug Cane Technol* 2: 195-200.

Ness JN (1984). Viscometry in cane sugar processing. *Proc Aust Soc Sug Cane Technol* 6: 271-277.

Rouillard EEA and Koenig MFS (1980). The viscosity of molasses and massecuites. *Proc S Afr Sug Technol Ass* 54: 89-92.