

THE USE OF CAPILLARY VISCOMETRY TO SURVEY VISCOSITY IN CANE SUGAR FACTORIES

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Abstract

Capillary viscometry is a simple and inexpensive technique that can be used to measure and compare the viscosities of syrups and of molasses. The technique was used to survey the dynamic viscosities of evaporator syrups, and A-, B- and C-molasses from four Tongaat-Hulett cane sugar factories throughout the 2006-2007 season. In addition, the effects of Brix, of purity and of temperature on the dynamic viscosity of these streams were investigated.

The survey highlighted process and climatic effects on the dynamic viscosities. It was also possible to investigate associations between high viscosities and poor factory performances. Exponential models were used to quantify the effects of Brix, of temperature and of purity on the dynamic viscosity of syrup and of molasses. Finally, an equation was derived to yield the dynamic viscosity of molasses given the Brix, purity and temperature. It was concluded that capillary viscometry can be a useful tool at the factories.

Keywords: sugar factory, viscosity, capillary, viscometry, survey, processing, dynamic viscosity

Introduction

In a recent publication (Lionnet and Pillay, 2006) it was shown that capillary viscometry is both precise and accurate for the measurement and comparison of the dynamic viscosity of juices and syrups. Furthermore, it was shown that it could be used to measure and compare the dynamic viscosities of molasses after dilution to Brix values of around 60%. It was noted that the equipment is inexpensive and that its use involves simple procedures.

During the 2006-2007 season, weekly composite samples of evaporator syrup, and A-, B- and C-molasses were collected at four Tongaat-Hulett factories (Maidstone, MS; Darnall, DL; Amatikulu, AK; Felixton, FX) and analysed for dynamic viscosity in the Technology and Engineering Group (TEG) laboratory, using procedures described by Lionnet and Pillay (2006).

In addition, the weekly composites from each mill were sub-sampled to prepare season composites for each material. These samples were used to investigate the effect of Brix, of temperature and of purity on the dynamic viscosity. In all cases, sugar solutions of the relevant Brix values and at the required temperatures were treated as standards; the dynamic viscosities of the standards were obtained from Bubnik *et al.* (1995).

Theory

When a liquid flows through a capillary 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 dynamic viscosity of the liquid being investigated with that of a standard, 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 dynamic viscosity of the liquid can then be obtained knowing the dynamic viscosity of the standard (James and Prichard, 1974).

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 dynamic 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:

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

Using the dynamic viscosity of the standard, that of the given liquid can be calculated. Temperatures must obviously be the same.

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 dynamic viscosities, it is necessary to have a number of viscometers with tubes of different radii.

Results

Trends at the factories

The weekly samples of syrups and molasses were diluted to Brix values of 40 and 60% respectively, and the dynamic viscosities determined at 20°C. Since Brix and temperature were kept constant for all the measurements, any changes in dynamic viscosity reflect differences in the compositions of the syrups and molasses; these seasonal trends, at each factory, are shown in Figure 1.

The following observations can be made from the results in Figure 1:

- Except at AK, dynamic viscosities at the factories were relatively steady from week 10 to week 31; this applies to syrup and to molasses. Thus, climatic and operational conditions at each factory, over the period May to September 2007, did not change sufficiently to affect viscosities.
- AK reported mechanical problems with a factory water cooling system over weeks 23 to 26; operating temperatures in the factory rose and there were visible signs that Maillard-type reactions occurred. The viscosity profiles at AK clearly show large increases (25 to 50%) over that period; viscosity increased in the syrup and in all the molasses, indicating that the reactions took place in the evaporation, pan boiling and C stations. It is evident that these reactions can cause large increases in dynamic viscosity. There are indications that the target purity difference (TPD) increased by 0.5 to 1.0 unit over that period.
- It is also interesting to note that, from May to September 2007, the dynamic viscosities were similar at the four factories. Again conditions on the North Coast were not sufficiently different to affect viscosity.
- The situation changed dramatically in week 32 (first week of October 2007). In all cases the dynamic viscosities increased, as shown in Table 1, where the viscosities have been averaged over the four factories. This continued until week 37 (10-16 November); thereafter viscosity decreased but did not return to the values seen at the beginning of the season. Dynamic viscosities for syrup cannot be compared to those for molasses since syrups were diluted to 40 Brix, while the molasses samples were diluted to 60 Brix.

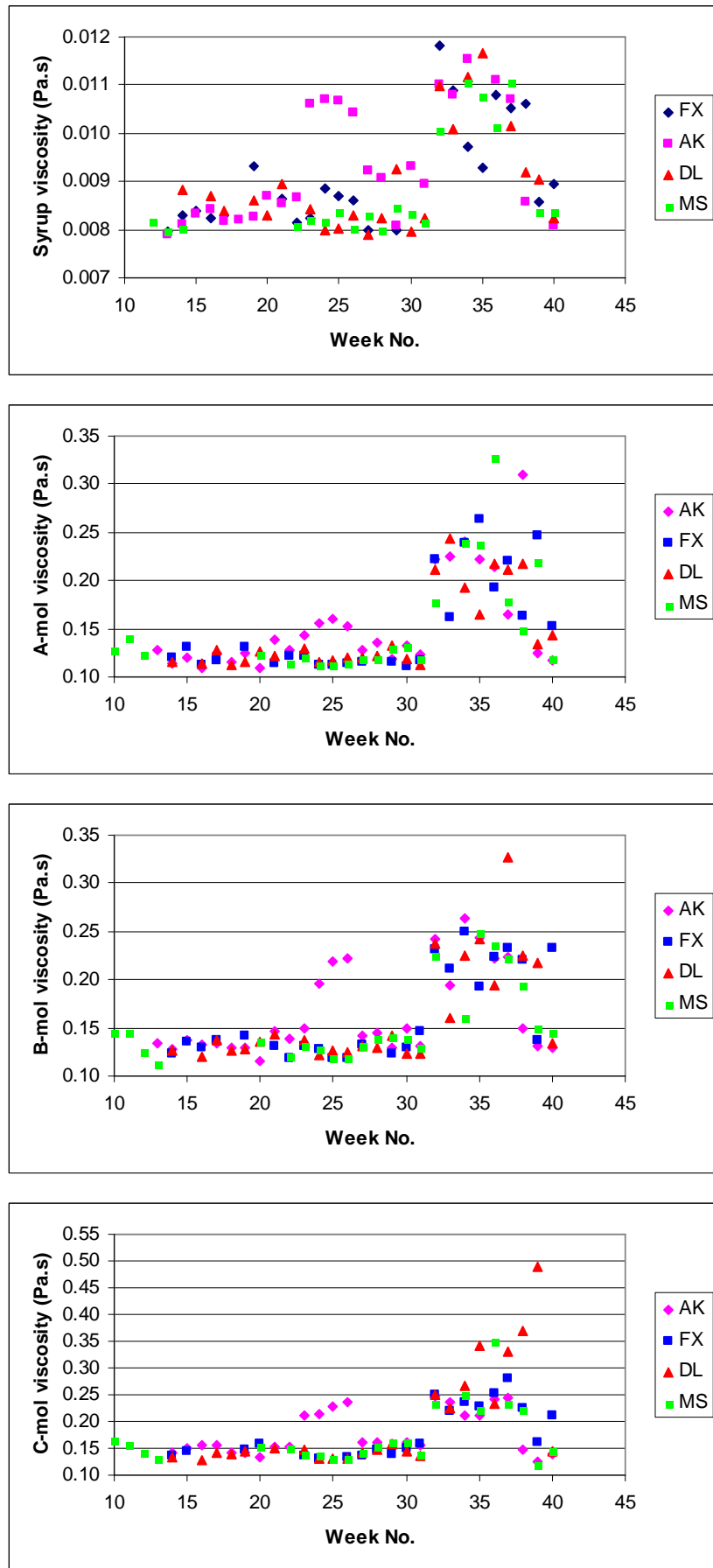


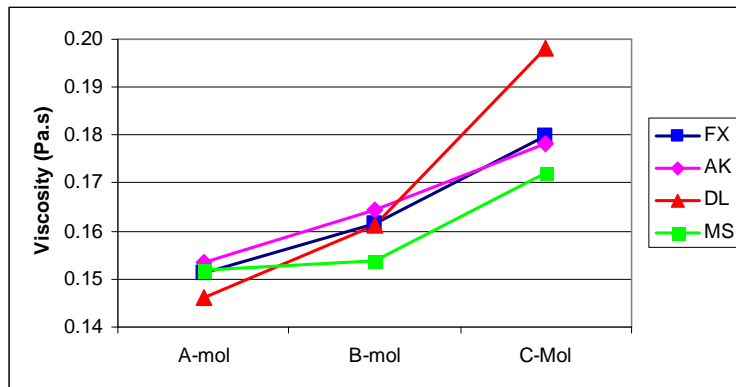
Figure 1. Trends in the dynamic viscosity of syrups at 40 Brix and of molasses at 60 Brix, measured at 20°C at four South African sugar factories during the 2006-2007 season.

Table 1. Dynamic viscosities (Pa.s) 2006-2007 season, averaged over four sugar factories in South Africa.

Material	May to Sept	Oct to mid Nov		Mid Nov to end Nov
	Viscosity	Viscosity	% change	Viscosity
Syrup	0.0084	0.0130	+55	0.0084
A-molasses	0.12	0.22	+83	0.17
B-molasses	0.14	0.23	+69	0.17
C-molasses	0.15	0.25	+67	0.21

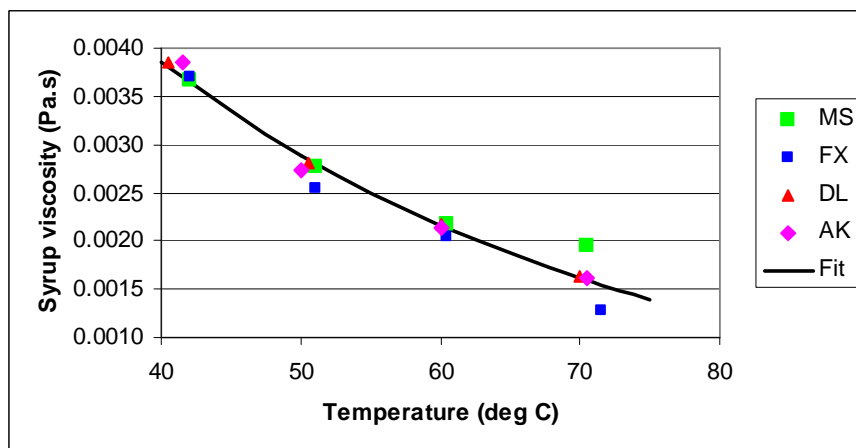
Clearly, climatic conditions can have severe and sudden effects on the viscosities of syrup and, more particularly, on that of molasses. Rain and heat are associated with poor cane quality and stop/start operations; this in turn negatively affects throughput, performance and sugar quality. All these factors interact and, as a result of this, it was not possible to estimate the impact of the viscosity increase on TPD.

Averaged dynamic viscosities for A-, B- and C-molasses, at each mill, have been plotted in Figure 2. The values are similar, with the largest difference being for C molasses. This difference, however, represents a gap of only 15%.

**Figure 2. Averaged dynamic viscosity of molasses at 60 Brix, measured at 20°C, at four South African sugar factories.**

Effect of temperature on dynamic viscosity

The weekly samples were used to prepare season composites of syrup and molasses, for each factory. These composites were then used to investigate the effect of temperature (40 to 70°C) on the dynamic viscosity of the syrups and molasses diluted to 40 and 60 Brix respectively, again using sugar solutions as standards. The results are shown in Figure 3.



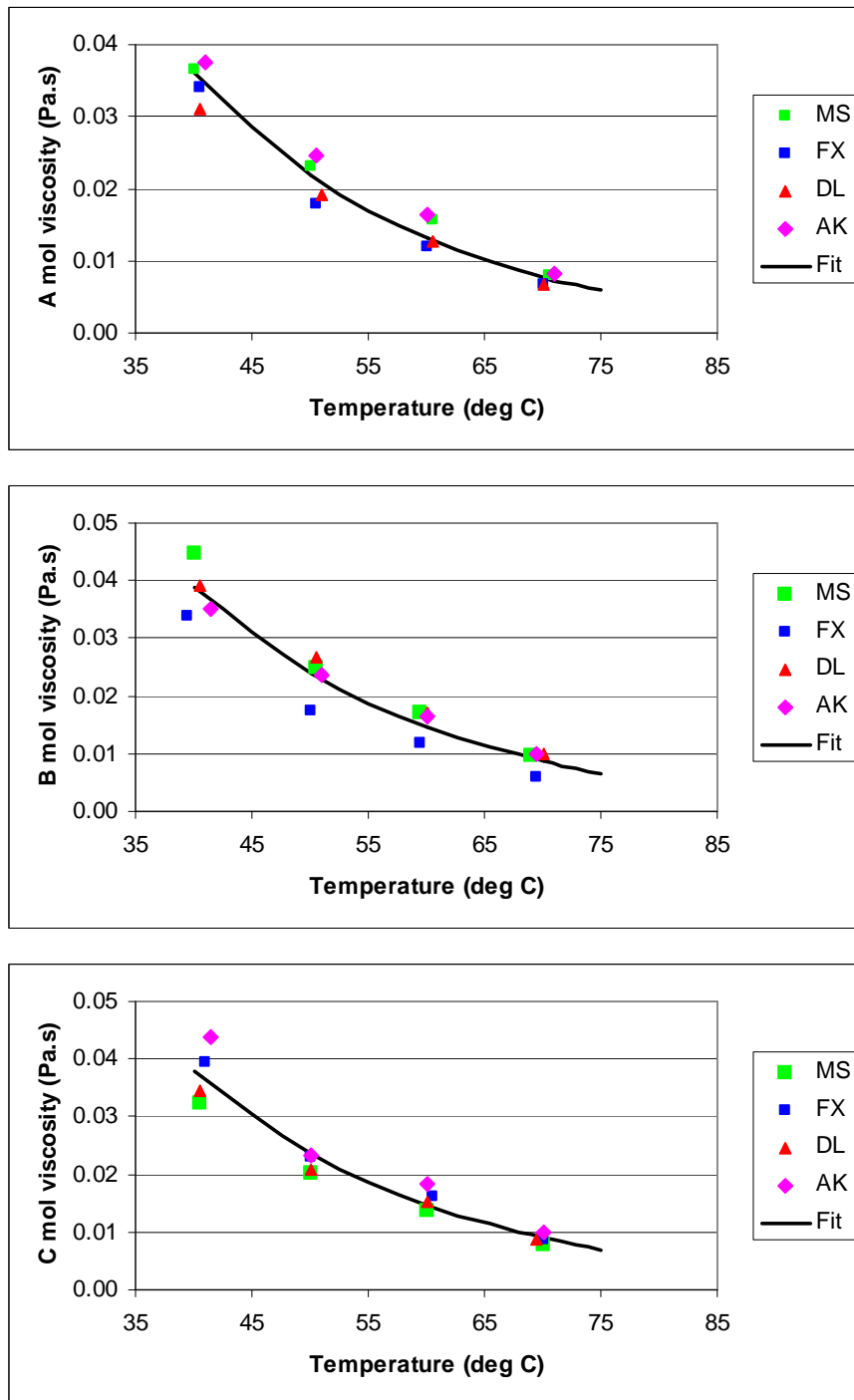


Figure 3. The effect of temperature on the dynamic viscosity of syrup (40°Bx) and of molasses (60°Bx) at four South African sugar factories. A regression line is fitted in each case.

Data are available for the dynamic viscosities of sucrose solutions over the temperature and Brix ranges used here (Bubnik *et al.*, 1995). These show that an exponential model describes the relation well. The fits in Figure 3 are of the form (equation 1)

$$\text{Viscosity} = a \times \text{Exp}(b \times \text{Temp}) \dots\dots(1)$$

where a and b are determined by least square regressions. The results obtained with the data presented in Figure 3 are given in Table 2; fits for pure sucrose have been included for comparison.

Table 2. Fits of exponential equations to model the effect of temperature (40 to 70°C) on the dynamic viscosity of syrups and of molasses.

Material	Brix (%)	Visc = a x Exp(b x Temp)		R ² (n=16)
		a	b	
Syrups	40	0.0123	-0.0290	0.94
Pure sucrose solution		0.00853	-0.0238	0.99
A-molasses	60	0.250	-0.0478	0.96
B-molasses		0.264	-0.0473	0.91
C-molasses		0.239	-0.0455	0.95
Pure sucrose solution		0.0955	-0.0372	0.99

Clearly, the fits are good and the simple exponential model can be used to quantify the effect of temperature on the dynamic viscosity of syrups and molasses at given, diluted Brix values. It is also evident that one equation could be made to fit the relevant molasses results from all the mills, except possibly for the FX B-molasses, but this has not been tested statistically.

Effect of Brix on dynamic viscosity

The composite samples were used to investigate the effect of Brix on the dynamic viscosities measured at 20°C, using sucrose solutions as standards. The ranges of Brix values were 30 to 60% for the syrups, and 48 to 60% for the molasses. As was the case for the study of temperature, data for sucrose solutions (Bubnik *et al.*, 1995) were used to investigate the type of relationship expected; the range of Brix values with the sucrose solutions was 48 to 70. It was immediately apparent that the simple exponential model, as used for temperature, gave poor fits, particularly at the higher Brix values where extrapolation is needed to estimate viscosities for factory materials. Consistent biases of -20 to -60% were found at the higher Brix values. Exponential models with increased curvature at the higher Brix values were tested; the following relation (equation 2) fitted the data well:

$$\text{Viscosity} = a \times \text{Brix} \times \text{Exp}(b \times \text{Brix}^c) \dots\dots(2)$$

The values of a, b and c were obtained by using graphical and numerical techniques, involving solver in Excel. This approach was tested with the data for pure sucrose solutions; the results are shown in Figure 4. The fit is good, particularly at the higher Brix values; the average error is now $\pm 5\%$, while the maximum error is $\pm 10\%$.

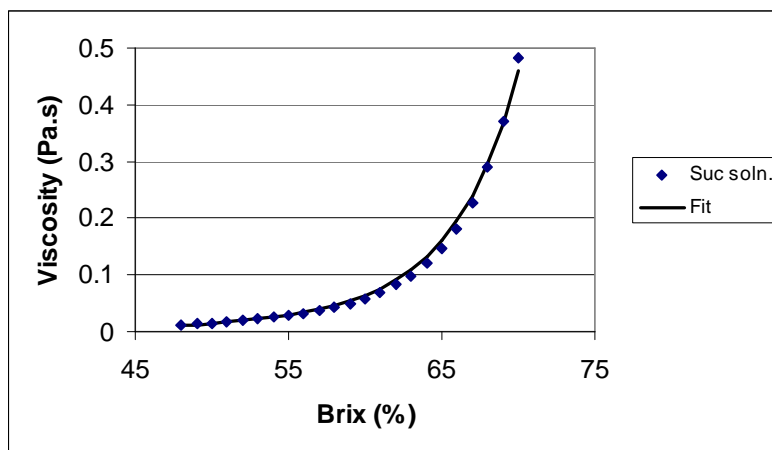
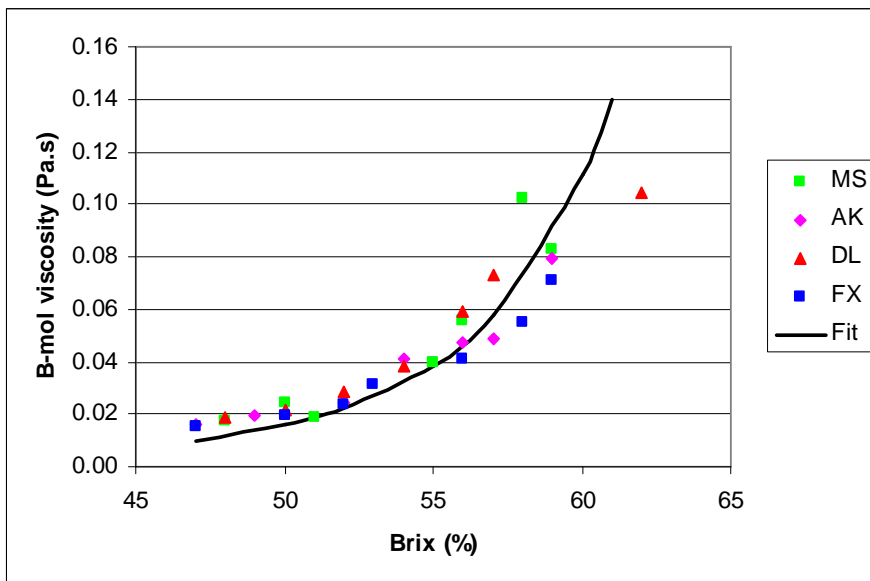
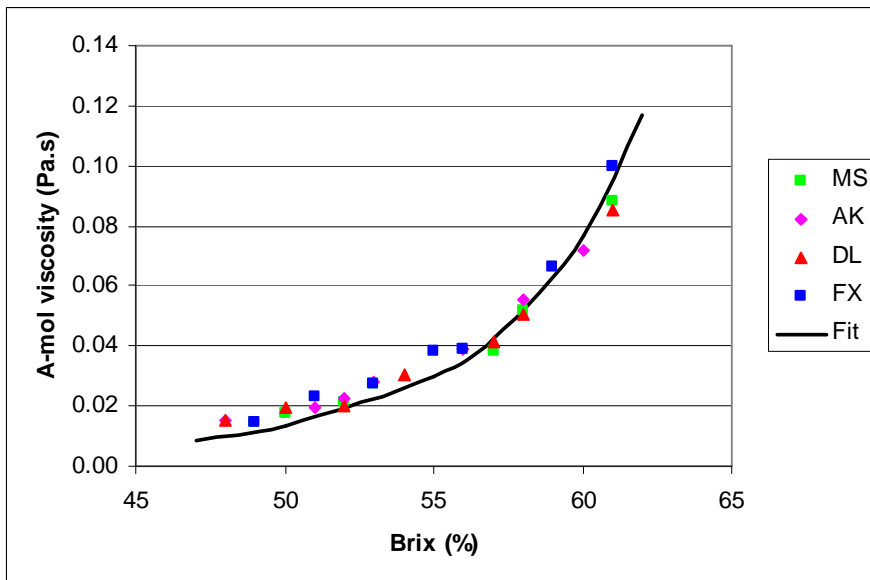
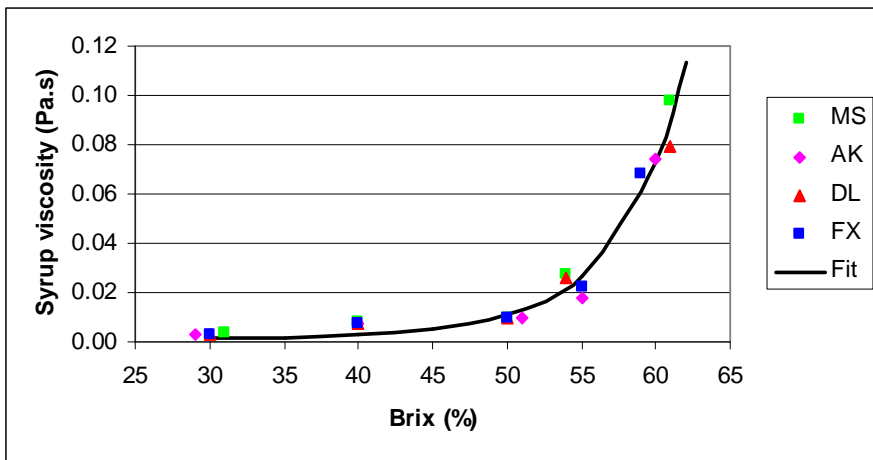


Figure 4. The fit of equation 2 to the dynamic viscosities of pure sucrose solutions at 20°C, over the Brix range of 48 to 70%.

Equation 2 was fitted to the data for syrups and molasses; the fits are shown graphically in Figure 5. There is some scatter but not much bias and, more importantly, the equation fits the data well at the higher Brix levels. This would allow meaningful extrapolation to factory Brix values.



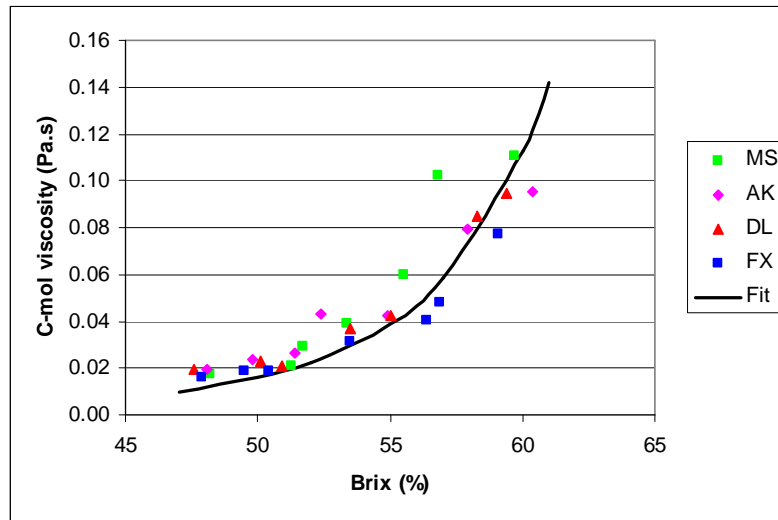


Figure 5. The fit of equation 2 to the dynamic viscosities of syrups and molasses at 20°C, over Brix ranges of 30-60% and 48-60% respectively, at four South African sugar factories.

The results obtained with the data presented in Figure 5 are given in Table 3.

Table 3. Fits of equation 2 to model the effect of Brix on the dynamic viscosity of syrups and of molasses.

Material	Brix range	Visc = a x Brix x Exp(b x Brix ^c)			Average error (%)
		a	b	c	
Syrups	30 to 60	2.50x10 ⁻⁵	1.19x10 ⁻⁵	3.10	±30
A-molasses	48 to 60	3.20x10 ⁻⁵	1.23x10 ⁻⁵	3.08	±15
B-molasses		3.00x10 ⁻⁵	1.32x10 ⁻⁵	3.09	±20
C-molasses		3.00x10 ⁻⁵	1.35x10 ⁻⁵	3.09	±20

Effect of purity on dynamic viscosity

The effect of purity on the dynamic viscosity was investigated on C-molasses only. Sugar was added to the various C-molasses samples in calculated quantities to yield a range of pol/Brix purities of 34 to 40; the samples were diluted to 60 Brix and the dynamic viscosities determined at 20°C. The results are shown in Figure 6. The simple exponential form used with temperature again fits the data; the relation (equation 3) is:

$$\text{Viscosity} = 0.534 \times \text{Exp}(-0.0417 \times \text{purity}) \dots\dots(3)$$

with an R² value of 0.74 for 16 pairs of observations.

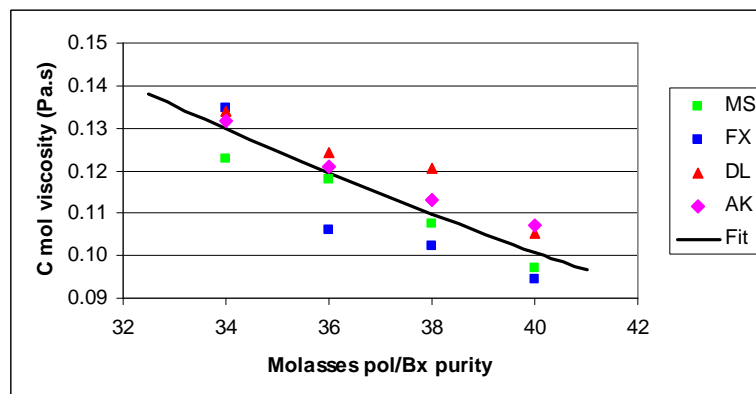


Figure 6. The effect of purity on the dynamic viscosity of C-molasses (60°Bx) at four South African sugar factories. A regression line has been fitted.

The impact of C-molasses viscosity on processing

Unless experimental procedures dealing with investigations in cane sugar factories have been rigorously planned to separate the interactive effects of such factors as climate, cane quality, and throughput, it is impossible to establish clear cause and effect between the factors investigated. The results available here are from a simple survey of the dynamic viscosities of syrups and molasses over time, at four factories; it is therefore impossible to determine any cause and effect relationships. At best, associations between factors may be highlighted.

Such an association was mentioned at AK, where visual evidence of Maillard-type reactions was associated with a sudden increase in viscosity and poorer TPD results. These changes were easier to note because they took place during a period when climatic and operational conditions at AK were steady.

The dynamic viscosities of C-molasses at each factory were averaged on a monthly basis. Monthly values for corrected reduced boiling house recoveries (CRB) and for the sum (sucrose lost in final molasses % sucrose in cane + sucrose lost in undetermined % sucrose in cane) were obtained for each factory. Any associations between the dynamic viscosity of C-molasses and these two performance parameters were then investigated. Evidence of associations was found at all the factories, except AK; the association was more evident between viscosity and the sum of the two losses. Two cases are shown in Figure 7.

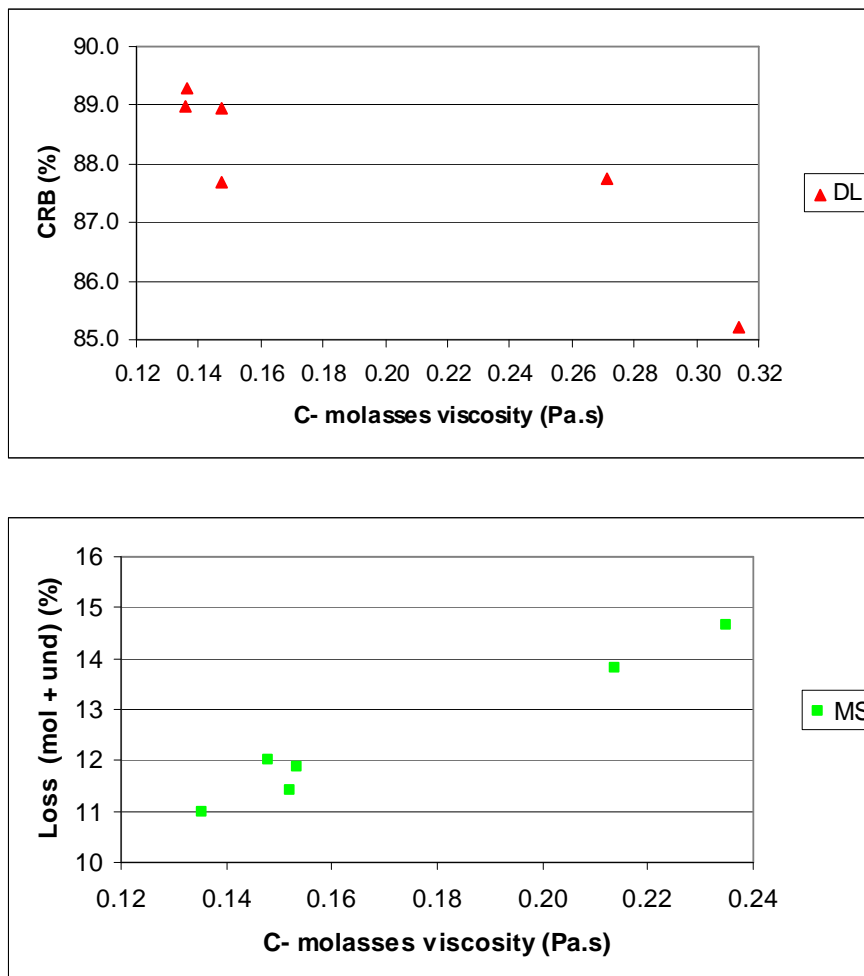


Figure 7. Associations between the dynamic viscosity of C-molasses and CRB at the Darnall factory, and sucrose losses at the Maidstone factory.

Molasses dynamic viscosity as a function of Brix, temperature and apparent purity

All the molasses data were used to investigate the possibility of obtaining one equation that would yield the dynamic viscosity of molasses (A, B or C) if the Brix, temperature and apparent purity were known. The natural logarithm of the dynamic viscosity was regressed against natural logarithms of Brix, temperature and apparent purity. This yielded equation 4:

$$\ln(\text{viscosity}) = -30.39 - 1.627 \times \ln(\text{temp}) + 8.325 \times \ln(\text{Brix}) - 0.310 \times \ln(\text{purity}) \quad \dots(4)$$

with an R^2 value of 0.93 for 148 pairs of observations. Dynamic viscosities calculated from equation 4 have been plotted versus the measured values in Figure 8. There is some scatter but no bias; the average error is $\pm 15\%$ while the maximum error is $\pm 67\%$. The ranges covered were 47 to 62% for Brix, 20 to 71°C for temperature and 30 to 75 for apparent purity.

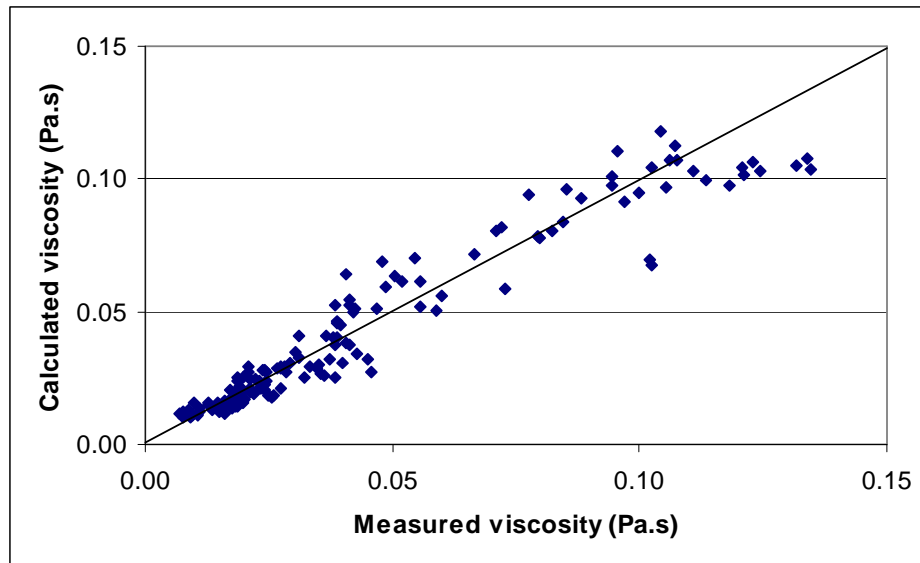


Figure 8. Viscosities calculated from equation 4 plotted against the corresponding measured values. The viscosities apply to A-, B- and C-molasses.

Discussion

The first objective of this work was to generate viscosity data on a weekly basis for each factory. The results obtained show that capillary viscometry as used here is well suited for this application. The technique is simple, inexpensive and rapid; using sucrose standards gives accurate values for the dynamic viscosities. Although the determinations were done at a central laboratory, there is no doubt that laboratory staff at the factories could easily deal with them.

The approach can highlight viscosity effects on performance, throughput and quality; this should help production staff justify and take corrective actions, if any. At one factory the trends of viscosity over time showed the impact of Maillard-type reactions on the viscosities of syrup and molasses, which was accompanied by poorer performances, particularly in terms of TPD. The association between high viscosity and poor TPD values has been presented previously by Koster *et al.* (1992). The survey also highlighted the sudden and pronounced effect of climate on viscosity, with increases of 70 to 80% in molasses viscosity, from one week to the next in October 2007. Again, this was accompanied by lower CRB values and higher sucrose losses.

The survey has also allowed the effects of Brix, of temperature and of purity on dynamic viscosity to be estimated, although this was not an original objective. This type of work has been a main objective of many authors (Barker, 1998; Broadfoot and Miller, 1990; Rouillard and Koenig, 1980); generally, however, these investigations have involved Brookfield rotating spindle viscometers and have been done in research institutions. An important limitation present here is that Brix and pol, rather than dry solids and sucrose, have been used. The results are, however, aimed at factory operations where dry solids and sucrose are not usually available. Exponential forms fitted the data well; generally, the effects were similar at the four factories. The average error between measured viscosity values and those calculated through the regression equations were $\pm 10\%$ and $\pm 20\%$ for the temperature and Brix effects, respectively. All the data for A-, B- and C-molasses were then combined and used to derive an overall equation relating the viscosity to Brix, temperature and purity; the logarithmic model used fitted the data well, with an average error of $\pm 15\%$. Rouillard and Koenig (1980) quote average errors of $\pm 20\%$ for this type of work but using more sophisticated approaches.

Conclusion

The main conclusion that arises from this investigation is the simplicity and usefulness of capillary viscometry as a tool that can be used in the laboratory or in factories to trend the viscosities of syrups and, more particularly, of molasses. Relationships between viscosity and performance parameters could be developed for a particular factory; it would then be possible to investigate the financial aspects of using chemicals such as sulphur dioxide or enzymes, of modifying the process, or of installing more equipment, to reduce the viscosity.

Acknowledgements

The author would like to thank laboratory personnel at the Amatikulu, Darnall, Felixton and Maidstone factories for the samples. Analytical Chemistry in-service trainees did all the experimental work at TEG; the author is particularly indebted to Sandisiwe Zondo and Pumeza Gedu.

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