A MODIFIED CONSTANT PRESSURE FILTERABILITY TEST FOR RAW SUGAR

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Abstract

The filtering properties of sugar liquors have been, and are, an important research topic. Two aspects need consideration; the first deals with the behaviour of the sugar during refining and the second is the actual mechanical measurement of the sugar liquor in the laboratory. Many laboratory methods are based on comparative, time dependent procedures. This paper describes a method based on a filtration theory developed for solutions containing small amounts of particles, as opposed to the conventional slurries. It is shown that the filtration of raw sugar solutions in the laboratory follows the proposed theory well. The test has been used to investigate the effects of factors such as the pH and temperature at which the filtration is done, the impact of the presence of residual polyacrylamide flocculant, that of suspended solids, and that of microbiological activity. Sugars from different factories and produced at different times of the year were used to investigate geographical and seasonal effects. Finally, further work, which includes the automation of the laboratory equipment, is suggested.

Keywords: filterability, sugar quality, suspended solids, filtration rates, pore resistance, filtration theory

Introduction

The filtering properties of sugar liquors have been, and are, an important topic for research. In South Africa for example, investigations started in 1957 with Alexander's classical paper; Douwes-Dekker contributed in 1964, Murray in 1972, 1974 and 1976. Morel du Boil produced a literature survey in 1997 while Simpson and Davis (1998) looked at the impact of various components on the filterability of raw sugars. Work has also been done in Australia (Crees *et al.*, 1977), in the USA (Vianna, 2000; Devereux and Clarke, 1984a), in the UK (Donovan and Lee, 1995) and in Japan (Yamane *et al.*, 1968).

There are two distinct aspects to the filtering properties of raw sugar liquors. The first is the behaviour of the raw sugar during refining; all refining processes involve a filtration/separation step at some stage of the operations.

The second aspect is the actual mechanical measurement of the filterability of sugar liquors in the laboratory. This usually requires a filtration apparatus, with filter aid and/or membranes, used under specified conditions. Nicholson and Horsley presented a constant pressure method in 1956 which was subsequently used and/or modified by a number of workers. Sullivan in 1970, among others, then introduced a more theoretical approach to the determination. In South Africa the Sugar Terminal (SAST) uses a method which is essentially the Nicholson and Horsley one. Ideally this laboratory determination of the filterability should give a good prediction of the behaviour of the sugar during refining.

More fundamental methods are based on filtration theories and usually require plots of filtrate volume against time. Other physical quantities such as viscosity, pressure differential and filtration area are required to calculate various filtration resistances. The results obtained are more general but are still influenced by the conditions under which the determination was done. The experimental procedures must therefore be clearly specified in all cases.

Theory

Sullivan (1970) proposed a method based on the relation between the filtrate volume and the filtration time, given by

$$\frac{t}{v} = bt + a \qquad (1)$$

where t is the filtration time and v the filtrate volume. Obviously this approach requires the plot of t/v versus t to be linear. The lower the value of b the better the filtration.

Murray (1976) used the same approach to investigate the filtration behaviour of South African raws. The results obtained by these two workers confirm that the filtration of sugar liquors follows the standard blocking equation of Hermans and Bredee, as discussed by Grace (1956). When the filterability is estimated not from the volume that flows during a certain time but rather from the continuous increase in resistance to flow as the volume of filtrate increases, one gets more leeway with respect to experimental conditions.

This approach requires that a plot of volume of filtrate (v) versus time (t) follows an asymptotic trend as represented by equation 1. Elementary algebra shows that, if equation 1 applies, then a plot of t/v against t yields a straight line, with slope equal to b and intercept equal to a. It is expected that the theory will use those two quantities to characterise the filtration and thus the properties of the original sugar. The intercept, a, gives an indication of the filtration rate; large values indicate low filtration rates. The plugging constant (PC, m^{-3}) is given (Grace, 1956) by the product a x 2. The slope, b, gives an indication of the final or asymptotic volume of filtrate. A large slope indicates a large final volume of filtrate. The pore resistance constant (PRC, m^{-1}) is given (Grace, 1956) by

$$PRC = \frac{a \times A \times \Delta P \times g}{\mu} \qquad (2)$$

where A is the filtration area in m^2, ΔP the pressure differential in Pa, g a dimensionless constant equal to 9.8, and μ the viscosity in Pas. PRC attempts to correct for some of the experimental factors and thus estimates the filtration properties of the sugar.

The selection of the values for the physical conditions to be used for the laboratory test has always been difficult and is usually a compromise. Ideally, the following should be addressed:

- The refining conditions, for example pH, temperature and Brix, should be used.
- The test procedures should be such that the measurements (volume, time, etc) can be made relatively easily.
- The results should characterise the sugar tested. Thus dilution, dissolution, pH and viscosity effects should be considered.
- The type of filter medium also needs to be considered.

Method

The method adopted at the Sugar Milling Research Institute (SMRI) used the following conditions:

- 30 Brix sugar solution (750 cm^3) at 70ºC, prepared immediately before the test.
- Filtering area of 0.0013 m^2.
- Differential pressure of 20530 Pa.
- Millipore 3.0 µm pore size (nominal) membrane, with a diameter of 47 mm. This is a hydrophilic, mixed cellulose acetate and nitrate filter having a high water flowrate. It has a temperature maximum of 75ºC.

The results obtained from a large number of raw sugars showed the required linearity for the plots of t/v versus t, which also confirms the findings of Sullivan (1970) and Murray (1976).

Apart from the calculation of the plugging constant and of the pore resistance constant, the results were used to calculate two other values:

- The volume of filtrate (m^3) after 3000 seconds, V_{3000}, as an indication of the asymptotic filtrate volume.
- A rate of filtration, DV/DT in m^3s^{-1}, given by differentiating equation 1, and averaging the values over t from 10 to 3000 s.

The viscosity needed for the PRC was obtained from a correlation proposed by Peacock (1995), for pure sucrose solutions. This approach may become less valid when the sugar contains high concentrations of impurities such as dextran.

Results

The results obtained from more than 70 raw sugars can be summarised as follows:

- The filtration of all the sugar solutions followed equation 1, with excellent linearity for the plots of t/v versus t (r squared values greater than 0.997 for at least 20 pairs of observations).
- When ΔP was varied, large effects were seen on all the parameters, except PRC.
- The same conclusion applies when the filtration area or the solution Brix was varied.
- Large variations were seen among the different raw sugars, indicating that raw sugar quality has an important impact.

Repeatability
The repeatability obtained with the membrane is shown in Table 1.

Table 1. Repeatability of the filtration test with a membrane.

Repeat	V_{3000} (m^3x10^{-4})	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
1	1.85	10580	9.2	5.7
2	2.04	9494	11.1	6.4
3	1.95	10074	7.9	6.0
4	1.96	10054	6.4	5.9
5	2.03	9612	9.8	6.3
Average	1.97	9963	8.9	6.1
95% Conf. int.	± 5%	± 5%	± 20%	± 6%

The repeatability is acceptable but it is felt that more improvements are possible. Visual examination of the membranes after a test shows evidence of poor sealing and of some breakthrough of the liquor. This is due to the use of old but available equipment; a more efficient filter has been constructed and will be tested in the near future.

Effect of temperature
The system was used to investigate the effect of temperature on the filtration behaviour of a raw sugar. Solutions of the sub samples of the sugar were filtered at different temperatures, under otherwise similar conditions. The results are in Table 2.

Table 2. The effect of temperature on filtration rate and on PRC.

Temperature (°C)	DV/DT ($m^3s^{-1}x10^{-8}$)	PRC ($m^{-1}x10^{10}$)
74.2	9.6	3.4
64.2	8.9	3.6
54.9	7.8	2.8
45.2	7.2	3.5
23.0	6.0	3.6

Temperature impacts on the filtration rate but not on PRC. The Arrhenius equation was used to quantify the effect of temperature on the filtration rate. The following result was obtained:

$$\ln \frac{DV}{DT} = -13.4 - \frac{96.3}{RT} \quad (3)$$

with a r^2 value of 0.99 for 5 pairs of observation. The plot is in Figure 1.

The activation energy can be calculated and is found to be equal to 8 kJ/mol. For transport processes (where the rate limiting step is either diffusion or viscosity) the literature (Mullin, 1993) quotes values of 8 to 20 kJ/mol; thus the effect of temperature on the filtration rate appears to be a physical one, probably on the viscosity of the solution. PRC is reasonably constant, indicating that the temperature has no effect on the impact of the impurities on the filtration rate.

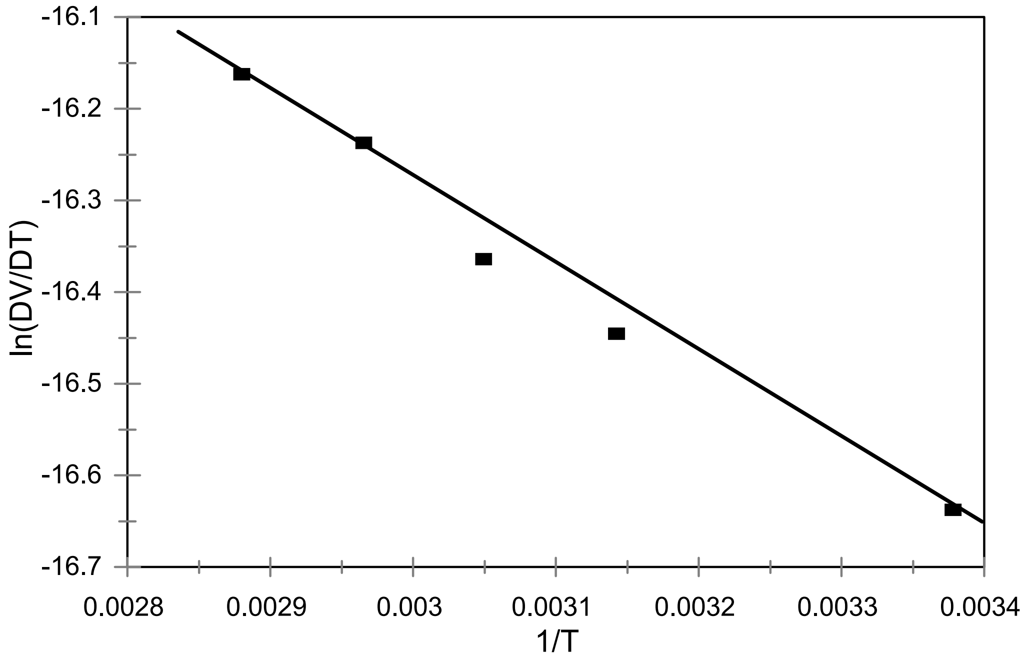

Figure 1. Arrhenius plot for the effect of temperature on the rate of filtration

Effect of residual flocculant

Magnafloc LT27, a high molecular weight, anionic flocculant was added to solutions of a raw sugar, and the filtration performance assessed. The results are in Table 3.

Table 3. Effect of a flocculant on filtration performance.

mg flocculant /kg sugar	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
0	7960	1.7	7.2
0.025	7068	1.7	8.2
0.25	10566	2.9	5.5
0.75	14284	4.5	4.1
1.25	14814	6.2	3.0
5	23868	8.3	2.5

Clearly, both the filtration rate and PRC are affected strongly by the flocculant. Concentrations around 0.025 mg per kg of sugar appear too low to cause changes, but concentrations above 0.25 mg/kg have a measurable effect. This test involved fresh flocculant, which would not be the case industrially.

Effect of pH

A triethanolamine buffer was used to change the pH of the sugar solution. pH correction is required in some of the filterability tests used industrially. The results obtained here are shown in Table 4.

Table 4. The impact of solution pH on filterability.

Feed solution pH	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
7.0	5520	11.0	11.0
7.0	6590	8.6	8.7
7.5	6432	6.1	8.5
8.0	5252	6.9	11.0
8.5	5560	6.5	10.0
9.0	11938	11.0	4.5
9.5	15588	13.0	3.4

Some of these results are shown graphically in Figures 2 and 3. The rate of filtration appears to be fairly constant up to a pH of 8.5 but then decreases sharply. The effect on the PRC seems more complex, with a minimum around pH values of 7.5 to 8.5. It increases at pH values above 8.5. It is probable that the pH effect depends both on the type and concentration of the impurities present in the sugar, and on the material used to correct the pH.

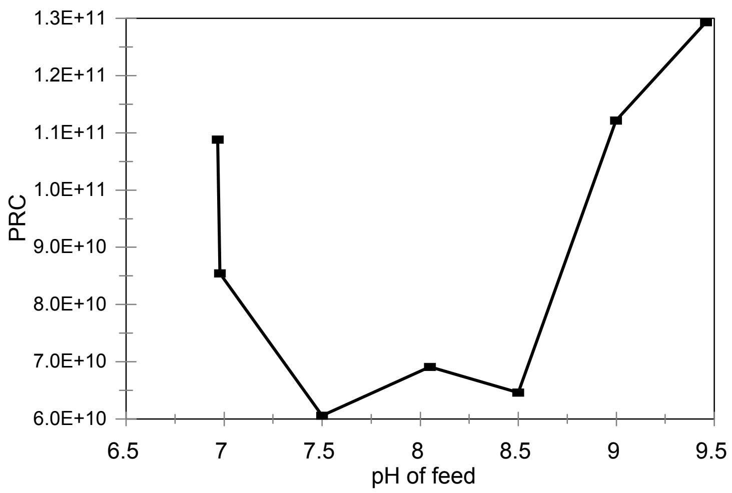

Figure 2. Effect of pH on PRC

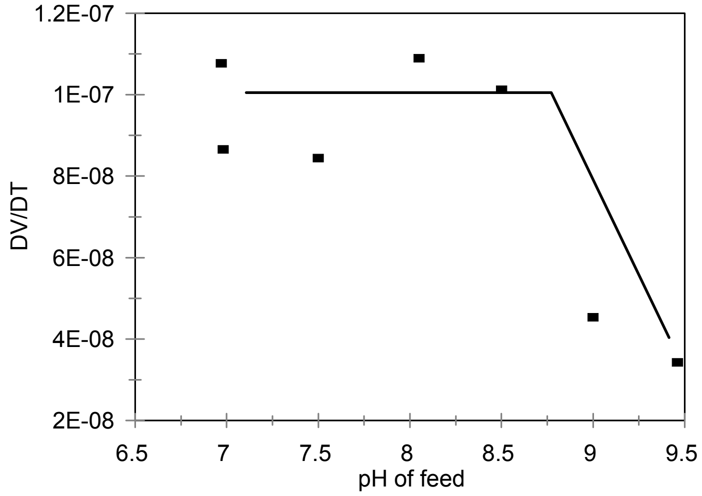

Figure 3. Effect of pH on the rate of filtration

Effect of suspended solids

The effect of suspended solids on the filtration behaviour of sugar solutions was investigated by testing the filtrate obtained through the 3 μm membrane. Thus the filtrate was treated as if it was a sugar solution. Filtrates from four very poorly filtering raw sugars were combined to obtain the required volume. Filterability data for the original sugars and for the combined filtrates are shown in Table 5.

Table 5. Results obtained with sugars and combined filtrates therefrom.

Material	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
Sugar 1	19910	2.6	2.6
Sugar 2	18206	4.2	3.2
Sugar 3	11864	3.2	4.9
Sugar 4	12484	3.3	4.6
Combined filtrates	5870	4.8	10.5

The volume of filtrate versus time relationships are shown in Figure 4. Finally, the results obtained with the above combined filtrates are compared to results from poor sugars and from the best raw sugar tested during this work, in Table 6.

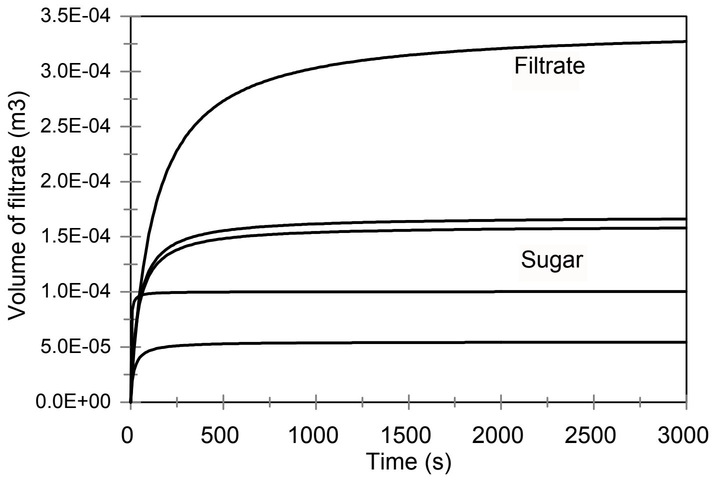

Figure 4. Filtration rates for original and filtered solutions

Table 6. Results from the combined filtrates, poor and best sugars.

Material	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
Poor sugar	15616	3.3	3.8
Best sugar	3710	N/A	16.1
Combined filtrates	5870	4.8	10.5

Clearly the first filtration through the 3 µm pore size membrane, has improved the behaviour of the solution considerably. The filtrates show performances which are now similar to those obtained with a very good raw sugar. This indicates that suspended solids have a marked impact on filterability.

Donovan and Lee (1995) also compared sugar solutions and the filtrates therefrom. They used 65 Brix solutions at 70ºC, filtered through Celite 505. All the filtrates showed improved filterabilities (expressed as %F) but the poorer sugars produced poorer filtrates. As was the case with the data shown in Table 6, the filtrates did not produce results which were as good as the best that could be expected.

Centrifuging instead of filtration, to remove the suspended solids, gave results which were less clear, but in all cases the filterability improved.

Clearly filterability is strongly associated with suspended solids.

Effect of microorganisms
The literature reports (Morel du Boil, 1997) that bacteria do affect the filterability of sugar solutions. They do so in three ways. The bacteria themselves block the filter pores; they produce soluble impurities that increase viscosity, and they produce colloidal gum like impurities which block the filter pores.

A 30 Brix sugar solution was tested immediately and subsamples allowed to stand for 2 and for 5 hours, at 30ºC before being tested again. The results are in Table 7.

Table 7. The effect of bacteria on filterability.

Standing time (hr)	V3000 (m^3)	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)
0	.00053	13	17
2	.00045	24	15
5	.00018	25	6

Clearly filtration rates have been affected negatively. The PRC values have also changed indicating a different impact from the impurities. These results indicate the possibility that microorganisms have been active, but this needs confirmation through bacterial counts.

Testing of VHP sugar intake at the South African Sugar Terminal (SAST)
Weekly VHP sugar samples from SAST, for the period October to December 2001 and originating from AK, DL, ES, FX, SZ and UK were tested. A monthly composite representing September 2001 was also included because no weekly samples were available for that period. Results averaged over each time period are shown in Table 8.

The relation between the filtration rate (DV/DT) and the SAST% filterability is shown in Figure 5. Clearly the two parameters are strongly related.

Table 8. Results for the SAST intake sugars.

Period	PC (m^{-3})	PRC ($m^{-1}x10^{10}$)	DV/DT ($m^3s^{-1}x10^{-8}$)	F(%) (SAST)
Sept 2001	10006	2.9	8.0	49
6/10	8757	3.2	8.0	52
13/10	10499	3.1	8.1	49
20/10	11116	2.4	6.7	36
27/10	14164	4.1	4.1	22
3/11	18991	8.1	3.5	23
10/11	12862	3.0	5.3	36
17/11	12846	3.1	5.5	35
24/11	25151	4.9	2.9	22
1/12	23846	5.0	2.7	18
8/12	22222	5.7	2.6	19

The trends for PRC and the filtration rate (DV/DT) are shown in Figures 6 and 7. PRC shows a peak at the end of October and an increasing trend at the end of the season. The filtration rate starts decreasing from mid October onwards. There are therefore strong seasonal effects.

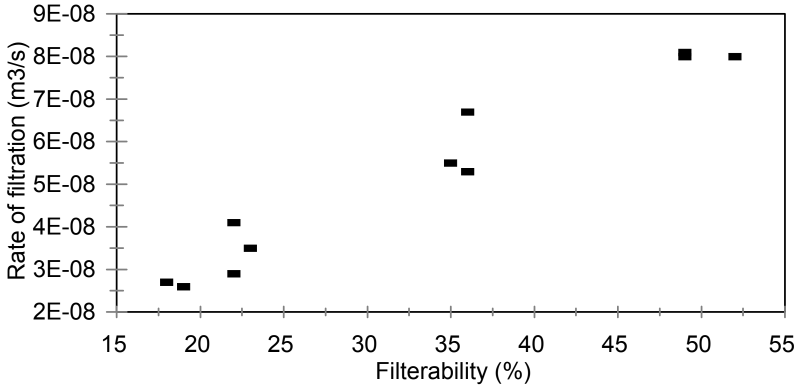

Figure 5. The relationship between DV/DT and percent filterability, SAST samples

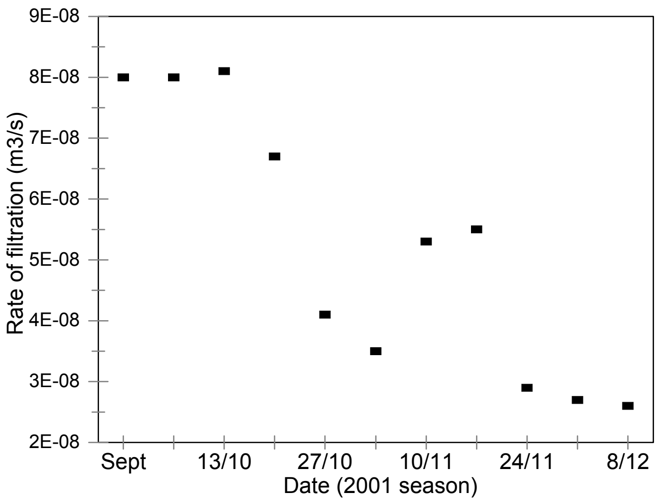

Figure 6. The trends in DV/DT for the SAST samples

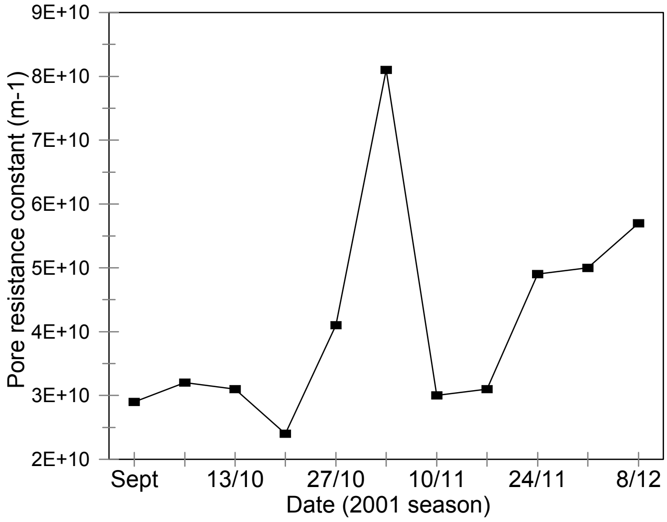

Figure 7. PRC values for the SAST samples

SAST also analyses the sugar samples for dextran (haze) and for starch. Since only a limited number of samples of sugar could be analysed by the SMRI filterability test, it is felt that the sample base is too small to allow meaningful analysis for the possible effects of these two impurities. Further work is required.

Comparison with results from other workers
Vianna (2000) provides data which allow the calculation of the pore resistance constant, of the plugging constant and of DV/DT. Eight sugars with pol % values ranging from 97.9 to 99.6 were tested. His results and those obtained with S African VHP sugars at the SMRI, are shown in Table 9.

Table 9. Results obtained in the USA compared to data from S African sugars.

	Vianna			**SMRI**		
	PC (m^{-3})	**PRC (m^{-1})**	**DV/DT (m^3s^{-1})**	**PC (m^{-3})**	**PRC (m^{-1})**	**DV/DT (m^3s^{-1})**
Minimum	12132	$10x10^{10}$	$0.3x10^{-8}$	3710	$1x10^{10}$	$1x10^{-8}$
Maximum	106094	$19x10^{10}$	$5x10^{-8}$	39434	$10x10^{10}$	$16x10^{-8}$
Average	34954	$16x10^{10}$	$2x10^{-8}$	14405	$5x10^{10}$	$6x10^{-8}$

Since the experimental conditions were not the same, (for example Vianna used filter aid), the results are not completely comparable. However they are not out of line and it should be possible to standardise the experimental conditions to improve the comparisons.

Analysis of suspended matter
A poor quality clear juice, in terms of turbidity, was sampled from the Sezela clarifier and centrifuged at 10000 g, for 15 minutes. The sludge was washed with water, centrifuged again and dried. The same procedure was carried out but using a solution of 30 Brix, made from a poor filtering VHP sugar, obtained from Darnall. The ashed sludges were analysed by X-ray fluorescence. The results are in Table 10.

Table 10. Analysis of ashed sludges from clear juice and from a VHP sugar.

Component (%m/m)	Clear juice	Poor filtering VHP sugar
Silica as SiO_2	20.8	50.8
Aluminium as Al_2O_3	13.0	14.2
Magnesium as MgO	2.4	1.6
Calcium as CaO	43.4	13.4
Phosphorous as P_2O_5	10.4	9.8
Iron as Fe_2O_3	7.1	7.8
Total	97.1	97.6

The losses on ignition were 82 and 77 % respectively, which include residual sucrose and moisture since the sludges were not dried to constant mass. At this stage the water/organic contents are high and have not been determined.

The results given in Table 10 apply to the inorganic material in the sludges. It is interesting to note that the same species account for most (> 97%) of the inorganic contents of the two sludges. Apart from silica and calcium the concentrations are similar in both sludges. Silicate accounts for half the amount of inorganic species in the sugar. Finally, clay is known to contain aluminium and silicates; it could thus be the source of the high aluminium and silica contents.

The sample of clear juice was a catch sample from Sezela, while the VHP sugar was a weekly sample from Darnall. These very preliminary results show that the species which are associated with poor filterability in sugar are similar to those found in the colloidal/suspended matter in clear juice. Clay, and thus soil or silt, could be an important component. Devereux and Clarke (1984b) confirmed the presence of soil in their study using electron microscopy.

This type of separation and analysis of the suspended matter should continue, with proper drying of the sludge, to allow the determination of both the inorganic and organic contents of the sludge.

Conclusions

An experimental method for the determination of the filterability of raw sugar has been developed, using basic filtration theory. It should allow better comparison between results obtained in different laboratories. It provides a rate of filtration and a parameter estimating the filtration resistance of the sugar.

The method is more complicated than the usual volume/time based determination used by SAST, but it could be automated. It is not intended as a replacement for that method but as an additional procedure to be used when somewhat more fundamental results are needed. It has been shown that the rate of filtration given by the modified method correlates well with the % filterability given by the comparative (SAST) method.

The method has been used to investigate a number of factors that could influence the filterability of VHP sugars. Two main results emerge. Firstly the effect of suspended solids, which is well documented in the literature, has been confirmed. The method will be used to carry out filtration through various pore size membranes in an attempt to investigate the size of the particles which are involved. Secondly, the sludge found in a poor filtering sugar and that from a turbid clear juice have been separated and analysed for their inorganic contents. These were found to be similar, with the presence of silica and of aluminium indicating that clay, possibly from soil carry over, could be associated with poor clarification and poor filterability. This type of approach, involving the separation and analysis of suspended matter, should continue.

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REFERENCES

Alexander, JB (1957). Some observations on the filterability of Natal raw sugars. *Proc S Afr Sug Technol Ass* 31: 68-75.

Crees, OL, Whayman, E and Willersdorf, AL (1977). Further studies on flocculation. *Proc Queensland Soc Sug Cane Technol* 44: 225-233.

Devereux, JA and Clarke, MA (1984a). Non sucrose components of cane sugar and efficiency of pressure filtration. *Proc Sug Ind Technol* 43: 36-59.

Devereux, JA and Clarke, MA (1984b). Observations on filtration impedance in raw sugar. Proc Sug Processing Research Conf: 209-230.

Donovan, M and Lee, FT (1995). Filterability of raw sugars - laboratory tests versus refinery performances. *Int Sug J* 97: 104-113.

Douwes-Dekker, K (1964). The filterability of raw sugars. *Proc Sug Ind Technol* 23: 159-170.

Grace, HP (1956). Structure and performance of filter media. *A I Ch E Journal* 2(3): 307-336.

Lee, FT and Donovan, M (1995). Filterability of raw sugars. *Proc Int Soc Sug Cane Technol* 22: 209-230.

Morel du Boil, PG (1997). Problems associated with refined sugar filtration. A survey of the literature. *Int Sug J* 99: 327-331.

Mullin, JW (1993). *Crystallisation.* Butterworths-Heineman Ltd, 3rd edition, London.

Murray, JP, Runggas, FM and Vanis, M (1974). Filtering quality of raw sugar: mechanism of starch influence on carbonatation. *Proc Int Soc Sug Cane Technol* 15: 1296-1307.

Murray, JP (1972). Filtering quality of raw sugar: influence of starch and insoluble suspended matter. *Proc S Afr Sug Technol Ass* 46: 116-132.

Murray, JP and Runggas, FM (1974). A study of factors influencing refinery carbonatation. *Proc S Afr Sug Technol Ass* 49: 90-93.

Murray, JP, Runggas, FM and Sheppard, GS (1976). Influence of raw sugar quality on the phosphatation process. *Proc S Afr Sug Technol Ass* 50: 179-183.

Nicholson, RI and Horsley, M (1956). The design and performance of a new test filter. *Proc Int Soc Sug Cane Technol* 9: 271-287.

Peacock, SD (1995). Selected physical properties of sugar factory process streams. Sugar Milling Research Institute Technical Report No. 1714. 46 pp.

Simpson, R and Davis, SB (1998). Investigations into the filtering quality of raw sugar. *Proc S Afr Sug Technol Ass* 72: 242-248.

Sullivan, JP (1970). Filterability tests using Millipore filters. *Proc Tech Session Cane Sug Refining Res*: 49-60.

Vianna, E (2000). Filterability study of raw sugar. *Proc Sug Proc Res Ins*: 253-255.

Yamane, T, Suzuki, K, Kaga, T and Takamizawa, Y (1968). The detrimental effects of impurities occluded in refined sugars on the sugar refining process. *Proc Int Soc Sug Cane Technol* 13: 380-384.