FILTERABILITY MEASUREMENT FOR RAW SUGAR QUALITY: THE TRANSITION TO A NEW TEST METHOD

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Abstract

Filterability of raw sugar is one of the foremost sugar quality parameters. To the refiner, the filterability of raw melt (the melted raw sugar) is important as this directly influences refinery throughput.

A study has been conducted by the SMRI to develop a laboratory filterability test method for raw sugars. The development of such a test and apparatus was requested by the South African sugar industry to enable factories to conduct their own analysis of raw sugar filterability, to allow problems to be detected early and corrective action to be taken by mill staff. The existing method, the South African Sugar Terminals (SAST) method, is only done at the Sugar Terminals; it cannot be done at the mills. Moreover, it has been found that the SAST method is not reproducible. Specifically, the SAST method uses filter aid, the test is done at cold room temperature, and the pH of the sample is adjusted to pH 9.0, all of which have been proven to influence the filterability result. The result is made worse by the long liquor sample standing time at these adverse conditions. Furthermore, it was also found that the SAST method under-predicts the filterability result of very good sugars and there is a potential that it may over-estimate the filterability of some poor sugars.

The filterability test developed by the SMRI is a modification of the pressure filter test. It is carried out on raw unaffinated sugar. The new filterability test makes use of easy to use apparatus that was specifically developed by the SMRI for this purpose. The method is quick and filtration is done at refinery temperature making it more representative of performance in a refinery and avoiding the negative consequences of low temperature on the filterability result. This new method was found to have good reproducibility compared to the SAST method as, firstly, it does not use filter aid but uses a specific membrane, which proved to give consistent results, and secondly, samples are filtered at their natural pH avoiding the influence of high pH on filterability result, and also avoiding the need for expensive buffer. The method holds true for all kinds of sugars from very good to very poor. This method, FilterKwik®, is proposed for adoption as the standard raw sugar filterability test method in South Africa.

Keywords: filterability, suspended solids, starch, sugar quality

Introduction

Filterability of raw sugar is one of the foremost sugar quality parameters. To the refiner, the filterability of raw melt (the melted raw sugar) is important as this directly influences refinery throughput. When a raw melt is difficult to filter, the refiner has to reduce the filtration Brix, and/or reduce the melt rate. Reducing the Brix gives a considerable reduction in viscosity and hence is quite effective at solving filtration problems, but this can be expensive in terms of the extra steam required to evaporate the excess water. If the evaporator cannot cope with the lower Brix, then the pan feed will have a lower Brix, slowing down the pans and causing a loss in production (Lee and Donovan, 1995).

A number of workers have studied filtration problems in sugar (Bennett, 1967; Bennett and Gardiner, 1967; Devereaux and Clarke, 1984; Donovan and Lee, 1994; Hidi and McCowage, 1984; Ramsey and Watts, 1974; Simpson and Davis, 1998; Simpson, 1999). The main reasons for filtration problems they found were suspended solids and starch. Suspended solids can affect all types of refineries, whether carbonatation, phosphatation or straight filtration (with a filter aid), whereas starch will mainly affect carbonatation by interfering with the growth of calcium carbonate crystals (Lee and Donovan, 1995). The mechanism by which suspended solids affect filterability is blocking of the pores in the cake and most of the problem is caused by particles less than 5 microns in size and generally less than 1 micron (Lee and Donovan, 1995). In South Africa, starch is generally well controlled primarily by the use of diffusion, but raw sugar mills which have a high starch problem will often also use alpha-amylase to reduce the starch levels. Suspended solids are therefore the predominant reason for poor filterability in South Africa, and indeed, the inspection of residues on the membranes has consistently shown that suspended solids are the main cause of filterability impedance.

A study has been conducted to develop a laboratory filterability test method for raw sugars that represents filtration performance in a refinery, and is also simple enough to be done at raw sugar factories in order to better control the filtration quality of raw sugar that the factories produce. The filterability test developed at SMRI is a modification of the pressure filter test and is carried out on raw unaffinated sugar. This method, FilterKwik®, and the modifications that led to it are described. This method will measure filtration problems caused by suspended solids, but, except for viscosity, this method will not indicate whether there will be a problem due to starch, as starch will not cause a filtration problem in this test method in the manner in which it affects carbonatation refineries, as is true for all the methods discussed in this report. As suspended solids is the main reason for poorly filtering South African raw sugars, this filterability test method is valid for the South African situation.

Materials and methods

The filterability test method

The need for a reliable laboratory filterability method for raw sugar quality evaluation has, in recent years, resulted in a search for a new test which is simple to operate and yields meaningful and reproducible results. The filterability apparatus developed by the SMRI is essentially a pressure filter and is shown in Figure 1.

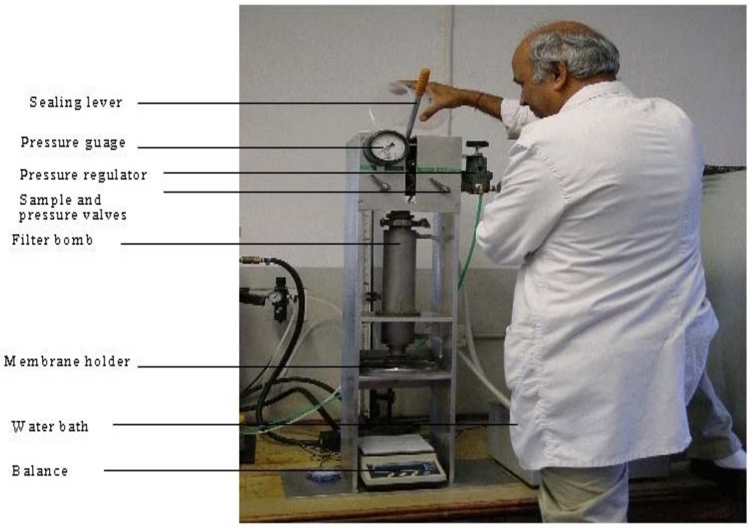

Figure 1. The filterability apparatus

Procedure for filterability test

- Prepare 600 grams of a 30 Brix sugar solution. No pH correction should be done. Cover and heat up to 70°C in a water bath.
- Assemble pressure filtration unit and then place the membrane on the screen support and seal the bottom of the filter unit.
- Place a beaker onto a scale to collect and weigh the filtrate.
- Circulate water through the jacket and maintain at 70°C.
- Once the sample reaches 70°C, transfer sample into the filter.
- Close the sample feeding valve and at the same time open the pressure supply valve to pressurise the filter to a preset and constant pressure (20.5 kPa or 3 psig).
- Simultaneously start the computer for logging. If there is no computer attached, start the stopwatch.
- Collect the filtrate and record mass and time. In case of computer logging, the computer does this recording automatically.
- The mass of filtrate collected after 45 minutes is used to calculate filterability.
- At the end of the run, rinse the filter well with hot water to remove any trace sugars and to prepare it for the next filtration.

Procedure for filterability calculation

The filterability determination based on a plugging value and the theory behind it has been described in detail by Lionnet and Ramsumer (2002) and the rate of filtration, DV/DT, is derived therein. It is felt that the high level of accuracy afforded by the detailed method is useful for research purposes but for factory use it is too elaborate, and where computer logging is not available it is simply not practical. It is proposed that filtration rate be derived from a single point measurement, i.e. the mass of filtrate after 45 minutes. All methods used commercially use a single point derivation of filterability.

Results and discussion

Expression of results

The initial proposal was to define % filterability as the mass of sample filtrate collected divided by the mass of filtrate of a standard refined sugar filtered under identical conditions. However, this was discarded after it was felt that refined sugar was too high a standard for sensible comparison to be made in this way (note that other methods used elsewhere use refined sugar as a benchmark sugar). Also, the starting mass of the sugar solution had to be much higher than 600 grams since refined sugar filters much faster. This would have meant a much bigger filter unit and larger sugar samples would have to be collected. This would have been too costly and disadvantageous where a large number of samples are handled as is likely in the sugar mills. Otherwise the time over which the filtration test is done would have to be shortened, and this would bring other disadvantages, as will be seen later. It was decided to find a very good raw sugar or its affinated equivalent that could be used as a standard. A sugar was found during the 2004/5 season from one of the South African mills which, under the standard conditions, filtered a total mass of 585 grams unaffinated. Since this was close to the starting mass of 600 grams, it was decided to simply use 600 grams of standard sugar solution as 100% filterability. Therefore, filterability is defined as:

$$\text{\% Filterability} = \frac{\text{Mass of filtrate of sample after 45 minutes (g)}}{\text{Total mass of sample fed into the filter (600 g)}} \times 100$$

In the case of sugars which are better than the benchmark, the effect will be to cap the filterability of such a sugar to 100%, which still indicates that the sugar is of very good quality. However, this is unlikely to happen, as it has not happened in the past three seasons.

Comparison of the new method with other methods

In Table 1, the new test is compared with other widely used test methods, namely: the Tate & Lyle Sugars test (Lee and Donovan, 1995), the South African Sugar Terminals (SAST) test and the Nicholson and Horsley (1956) test in order to highlight any differences. The SMRI method, FilterKwik®, is primarily compared with the SAST method, since this is the method used currently in South Africa. The differences in the test methods are important as shall be seen later.

Table 1. Comparison of SMRI test with other test methods.

	SMRI test (FilterKwik®)	Tate and Lyle test	SAST test	Nicholson and Horsley test
Liquor brix	30	65	60	60
pH	Natural	Natural	9.0 buffered	9.0 buffered
Temperature (˚C)	70	70	Room	Room
Pressure (psig)	3	60	50	50
Filter paper	3.0 µm membrane	Whatman GFA	Whatman No. 54	Whatman No.1
Filter aid	None	0.5% Celite 505	0.5% Celite 505	0.5% Celite 505
Liquor preheat	None	45 min	None	None
Standing time	None	3 min	15 min	None
Volume used for filterability	Filtrate in 45 min	Filtrate in 5 min	Filtrate between 2 and 7 min	Filtrate between 2 and 8 min

The SA Sugar Terminal determines filterability on all raw sugar cargoes it receives, and hence it was proposed to compare the SAST and the SMRI methods and check for a usable correlation between these methods. This study was initiated in 2001 by Lionnet and Ramsumer (2002) and a correlation reported (Old correlation in Figure 2). The study continued in the past season and it can be seen from Figure 2 that the past season's correlation (New correlation in Figure 2) is very different from that of 2002. The old correlation is believed to be disadvantaged by the fact that it compares too few sugars and these sugars were poor to average sugars. Good and very good sugars were not included in the correlation. For this reason, the new correlation is believed to be more correct. Furthermore, the latest data points, represented by triangles in Figure 2, seem to validate the new correlation, although there is a lot of scatter. The scatter is believed to be introduced by the inherent deficiencies in the SAST method, which are discussed below.

Choice of operating conditions

The main advantage of the SMRI method is that it is simple and its results are reproducible. Specifically:

- It does not use filter aid. It has been shown in previous studies ([1]unpublished data) with Celite as the filter aid that the results were not reproducible because the pad could not be produced evenly all the time. The use of a filter aid was eventually abandoned.

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[1] Lionnet R and Ramsumer R, SMRI

- The filtration test of the SMRI method is done at 70°C. This was done after several trials at room temperature resulted in results that could not be reproduced. It was later discovered ([2]unpublished data) that bacterial infestation of the samples at room temperature is very high. The bacteria would then interfere with filtration by blocking the filter pores. A temperature of 70°C also more closely reflects refining conditions. Temperature sensitivity like the pressure sensitivity test mentioned below was not done. This is because temperature in the new test is directly controlled automatically and sample behaviour does not influence the control of the temperature, unlike pressure.

- It has been found that raising the sample pH to 9.0 will give an incorrect view of the filterability of the sugar because of the reaction of the calcium acetate buffer used for pH correction with the residual phosphate in the sample which forms a precipitate of calcium phosphate (Simpson, 1999) lowering the filterability of the sample. Lionnet and Ramsumer (2002) also found that as the pH of the sample was adjusted the rate of filtration did not seem to change until the pH reached a value of about 8.5; thereafter it decreased abruptly.

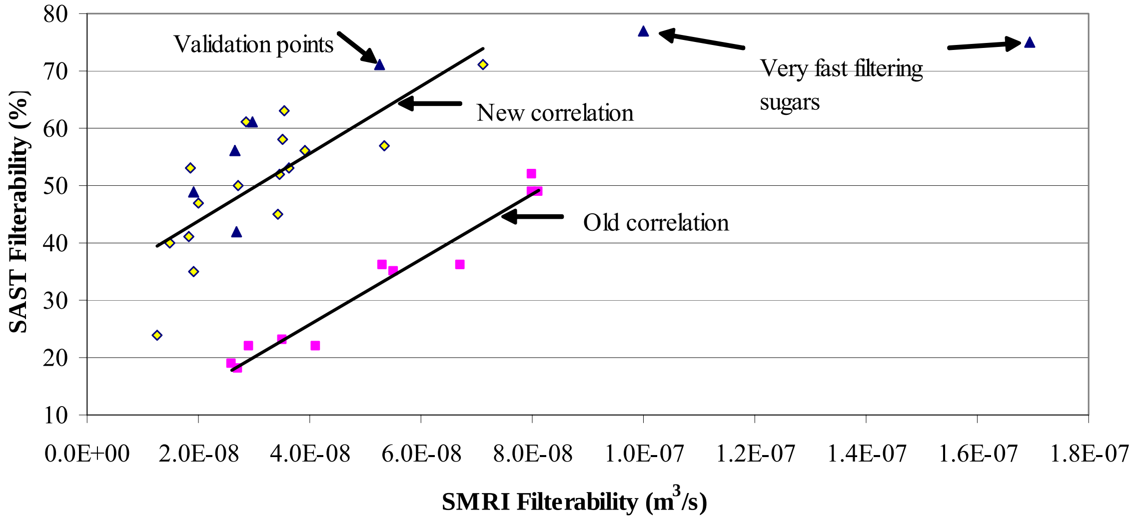

Figure 2. Comparison of the SAST and the SMRI filterability methods.

- Allowing solutions to stand for a long time, especially at high pH such as pH 9.0 and/or low temperatures such as room temperature, also results in an altered filterability result as noted above. The SMRI method has overcome this by having no standing time during the test.

- Whatman filter paper was tried but gave inconsistent results. These were resolved when a specific membrane was used instead of filter paper (Lionnet and Ramsumer, 2002).

- Control of pressure has been found to be satisfactory. All the methods noted above use automatically regulated constant pressure filtration, as is done in refining. While pressure regulation is important in these tests, a test was run in 2003 to check the pressure sensitivity of the filterability index. The pressure set point was changed ±2 kPa, i.e. a 10% error, and the variations in the results were found to be small (Davis and Ramsumer, 2003) and considered to be within the repeatability tolerances. A 10% error in pressure regulation is therefore considered acceptable.

[2] Lionnet R and Ramsumer R, SMRI

- It was also found that the linear correlation between the SAST and the SMRI methods did not hold for very fast filtering sugars as can be seen from Figure 2. A plot of a selection of below average to very fast filtering sugars (see Figure 3) revealed that the method used by SAST was not able to predict accurately the filtration behaviour of very fast filtering sugars. The SAST method under-predicted the filterability of the very fast filtering sugar (e.g. sample 4 in Figure 3) that was still producing filtrate after seven minutes (420 seconds). This will be a common problem with all methods that measure the filterability in the early part of the filtration time. A longer time would give a better approximation of the filtration rate for these sugars. The 45 minutes test time of the SMRI method accounts comfortably for these fast filtering sugars.

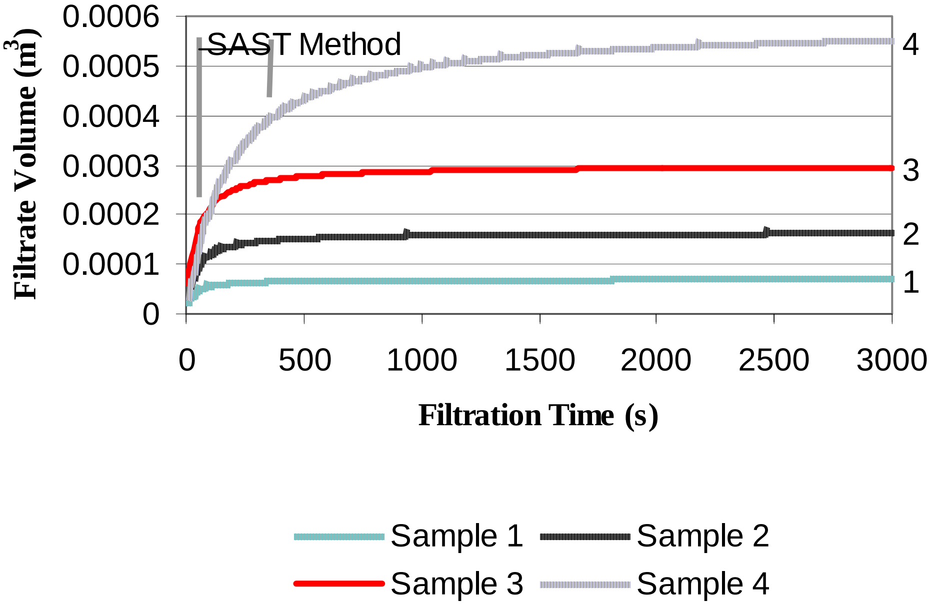

Figure 3. Comparison of filtrate volumes for four sugars.

Another important thing to note from this example is that the very good sugars do not necessarily start off with a fast rate of filtration, something which could lead to an under-estimation of the filterability of these sugars by methods that determine filterability from the initial filtration behaviour of the sugar. On the other hand, poor sugars that start off with a fast rate of filtration but do not keep it up could be mistaken for good sugars. It can be seen that a determination of filterability that covers a longer time span will give a more consistent result since the very good sugars consistently produce (over a long time span) a very large volume of filtrate.

Lionnet and Ramsumer (2002) also established this fact when they compared the filtration rates of poor sugars with those of filtrates that had already been filtered through a membrane (i.e. sugar solutions which have had suspended solids removed by the first filtration). The filtrate filtration did not necessarily have a fast initial rate of filtration (because of other factors such as viscosity), as can be seen from Figure 4, with some of the average sugars being better than filtrate, but it undisputedly resulted in an overwhelmingly larger filtrate volume after 45 minutes. Filterability determined from a long filtration time gives a more accurate reflection of the filtering quality of the sugar. It is also very important to note that filterability figures derived from early and shorter times are more prone to errors of observation and such errors may be considerable in these small volumes of filtrate.

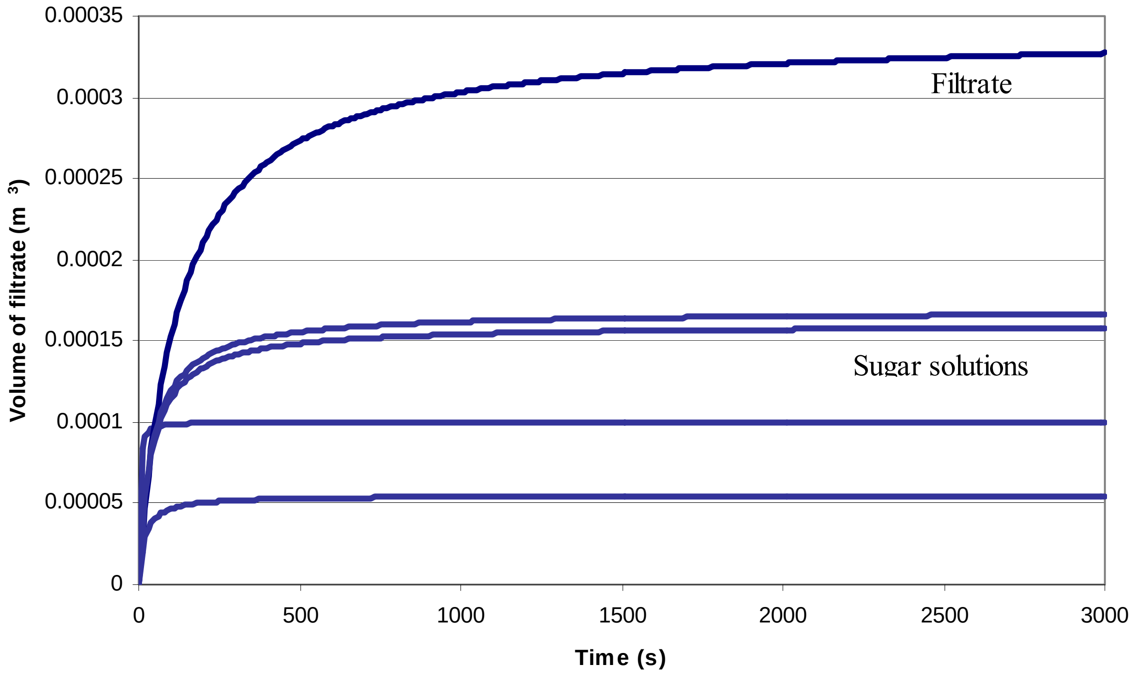

Figure 4. Filtration rates for original and filtered solutions.

For these reasons, it is believed that while there may be some directly proportional relationship between the SMRI (FilterKwik®) and the SAST methods (and for that matter with all the above mentioned methods), there can be no accurate correlation derivable from the SAST method and FilterKwik®. With all of the above differences considered, FilterKwik® will produce more accurate results which will correlate with and serve as a prediction of the actual behaviour of the sugar in a refinery.

Reproducibility test of FilterKwik®

Reproducibility trials were done on the FilterKwik® method and the following results (Table 2) were found. These were considered acceptable since differences between the sugars were found to be large in comparison. It is also proposed that the filterability tests be done in duplicate and the filterability values be an average of the two tests.

Table 2. FilterKwik® reproducibility test results.

Parameter	Test No.			Agreement
	1	2	3	
DV/DT (m^3s^{-1})	1.85 x 10^{-07}	2.05 x 10^{-07}	1.85 x 10^{-07}	±14%
V3000 (m^3)	3.54 x 10^{-04}	3.44 x 10^{-04}	3.59 x 10^{-04}	±5%
Plugging Constant (PC) (m^{-3})	5452	5664	5378	±6%
Pore Resistance Constant (PRC) ($m^{-1}s^{-1}$)	3.88 x 10^{10}	3.03 x 10^{10}	3.89 x 10^{10}	±32%

Notes: Agreement is defined as the relative mean deviation, which is the sum of all deviations from the mean divided by the mean. The mean is the arithmetic mean.

The definitions of these parameters are given in Appendix I.

It must be noted that when sub-sampling of the sugar sample is required, riffling must be used, otherwise there can be large differences in the results. Also, membranes have to be uniform to prevent the introduction of large errors, hence the reason for recommending a specific membrane.

Cost implications for changing to the new method

It is difficult to conduct a cost benefit analysis for the change since there is no accurately quantifiable benefit from the use of this test method. However, if it is assumed that the filtering quality of raw sugar produced by SA mills improves as a result of the test method being used at the mills, the benefit for this method may be taken as better throughput in both SA refineries and overseas refineries. This will result in lower cost of refining and for overseas refineries who are very particular about sugar quality that gives superior throughput, an improved filterability will assist with guaranteeing sales of export sugar to premier markets. The export sugar makes up about 50% of total sugar produced in SA, which works out to approximately 1.1 million tons per annum. Weighing that up against a cost of R584 000 for rolling out the new apparatus to all the mills, Hulref and SAST, the payback period could be a matter of weeks.

There are also cost savings from the present method. The new method is quicker, taking only 45 minutes compared to 2 hours for the present method, when pre-preparation time is considered. The analyst time should be cut by half, which should translate into human resource cost saving or better productivity.

Conclusions

A new filterability test method, FilterKwik®, has been developed.

- The method is essentially a pressure filter method but with enhancements that result in a robust method that produces reproducible results.
- The method is a great simplification over the present method and can be used in the sugar mill environment.
- While the filterability parameter determined by FilterKwik® is a considerable departure from the present one, the results are more accurate and will correlate with and serve as a prediction of the actual behaviour of the sugar in a refinery. The numerous disadvantages of the present method more than justify the need for change.
- In addition to the filterability result, inspection of residue on membranes can be used to identify the cause for poor filtration.
- The SMRI should provide a backup service in the event of unusual cases.

Recommendations

- It is recommended that FilterKwik® be adopted as a new filterability test method in South Africa, with a transition period of one season.
- It is recommended that during this transition period, both the SAST method and FilterKwik® be run in parallel and results from both methods be reported to introduce the new method to customers. It is also anticipated that during this transition period the specification and the penalty lines in the new method will be established. It is recommended that in the season thereafter, FilterKwik® be adopted as a standard method.
- A control sugar must be made available by the SMRI to the mills to periodically run checks on the calibration of the filter.

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APPENDIX I

The following parameters are defined in full by Lionnet and Ramsumer (2002). They are:

- DV/DT (m^3s^{-1}) is the rate of filtration which is obtained by averaging the values DV over DT from 10 to 3000s. Where DV is the change in filtrate volume and DT is the change in filtration time.

- V3000 (m^3) is the volume of filtrate (m^3) after 3000 seconds which is an indication of the asymptotic filtrate volume.

- PC (m^{-3}) is the plugging constant which is ($a\times2$) where 'a' is the intercept obtained from a plot of T/V against T which should be a straight line. V is the filtrate volume in m^3 and T is filtration time in seconds. The intercept, a, gives an indication of the filtration rate; large values indicate low filtration rates.

- PRC ($m^{-1}s^{-1}$) is the pore resistance constant given by

$$PRC = \frac{a \times A \times \Delta P \times g}{\mu}$$ where A is the filtration area in m^2, ΔP

is 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.