

A SEASONAL EVALUATION OF THE FILTERKWIK RAW SUGAR FILTERABILITY TEST

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Abstract

The filtering characteristics of raw sugar destined for refining have been the topic of much research. Investigations in South Africa on the impact of various components of raw sugar on the filterability characteristics of raw sugars started as far back as 1954.

Worldwide, there is still no uniform method to determine the filterability of raw sugars for use in export agreements. To address this, the Sugar Milling Research Institute developed a method, called FilterKwik, specifically to predict the behaviour of raw sugar during the first stages of refining. The aim was for the method to be representative of general South African refinery conditions so that it could be used to rapidly assess the suitability of a raw sugar for refining, be it local or abroad, bearing in mind that different refinery processes may be sensitive to different raw sugar quality requirements.

The FilterKwik method was earmarked to replace the SA Sugar Terminal method, in recognition of the value that this would add to the South African Sugar Association marketing strategy, pending a seasonal comparison study. This study was completed during the 2009/10 season when just under 400 raw sugar samples were subjected to duplicate analyses using the FilterKwik method as well as a range of other sugar quality tests typically performed by the Sugar Terminal laboratory. This paper reports on the seasonal evaluation.

Keywords: raw sugar, filterability, refining, export, SA Sugar Terminal

Introduction

The filterability of sugar as a quality indicator to grade raw sugars destined for refining has attracted a great deal of research over the years. Investigations in South Africa started as far back as 1954 (Alexander, 1957) and culminated in a paper by Simpson and Davis (1998) presenting a considerable amount of work done on the impact of various components of raw sugar on the filterability characteristics of South African raw sugars. (Also see review paper by Morel du Boil (1997).) Elsewhere, work has been done in Australia (Crees *et al.*, 1977), in the USA (Devereux and Clarke, 1984; Vianna, 2000), in the UK (Donovan and Lee, 1995) and in Japan (Yamane *et al.*, 1968). It is generally agreed that filterability is a sporadic problem that is caused by more than one factor and, usually, a combination of several factors (Lionnet, 2002).

According to Lionnet (2002), there are two distinct aspects to the filtering properties of raw sugars. The first is the behaviour of the melted raw sugar during refining, since all refining

processes involve a filtration/separation step. The second is the actual mechanical measurement of the filtering characteristics of the sugar in the laboratory. Ideally the laboratory method should accurately predict the behaviour of the sugar during the refinery separation step.

The SA Sugar Terminal (SAST) introduced a filterability test during the 1999 season specifically for segregation of sugars into poor, medium and good filtering sugars, destined for markets with specific requirements. The method chosen was a constant pressure method developed by Nicholson and Horsley (1956) but modified as considered for adoption by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) in 1966 (Anon, 1966; Anon, 2002). This will be referred to as the SAST method in this paper.

More fundamental methods are based on filtration theories, taking the physical characteristics of the solutions, such as viscosity, pressure differential and filtration area into account and usually requiring plots of filtrate volume against time. One such method was published by Sullivan (1970) and used by the Sugar Milling Research Institute (SMRI) as a basis for the development of their own method over a period of more than a decade (Lionnet, 2002). The idea was for the method to be representative of general South African refinery conditions, where possible, with specific emphasis on the effect of suspended solids. Since these solids are the main cause of poor filtration behaviour in South African raw sugars, this method would be used as an expedient refinery processing aid, bearing in mind that different refinery processes are necessarily sensitive to different raw sugar quality measures.

The initial procedure included measurement of the filtration rate from which the plugging constant, the final or asymptotic filtrate volume and the pore resistance constant could be derived. While use of the pore resistance constant could compensate for some experimental factors in the procedure, it was found that the final filtrate volume characterised the sugar just as well for factory operation purposes, which do not generally require the high level of accuracy and information assimilation needed during research. The outcome of the method was therefore simplified into a single value relating to the final filtrate volume only, although filtration rate data is still collected and stored (unpublished data¹).

After presentation of the SMRI method to the SA Sugar Industry (Lionnet, 2002), the method was earmarked to replace the SAST method, in recognition of the value that this would add to the marketing strategy of the South African Sugar Association. The method has since undergone rigorous testing and optimisation to constrict the reproducibility of the procedure. The most important change was the introduction of an 8 μm membrane (instead of 3 μm pore size) to render the method less sensitive to sample representivity (unpublished data²). This paper presents results from a seasonal evaluation of samples received at the SAST on a weekly basis during the 2009/10 season from May (week 12) to November (week 39).

Experimental

Filterability methods

The FilterKwik method is based on a constant pressure filterability apparatus that was developed by the SMRI (Lionnet, 2002). The output of the method was simplified into a single parameter, termed FilterKwik filterability (measured in %), as this value was sufficient

¹SMRI Technical Report No 1956. Mkhize *et al* – 21 February 2005

² SMRI Technical Report No 2076, Muir – 11 January 2010

for general classification of the sugar in terms of quality. A total of 210 raw sugar samples from 11 South African raw sugar factories, received weekly at the SAST, and a further 29 composite raw sugar samples were analysed in duplicate between May and November of 2009. SAST accreditation control samples (152 in total) were also analysed during this time.

The main differences in operating conditions between the FilterKwik and SAST methods are shown in Table 1.

Table 1. Operating conditions for the FilterKwik and SAST filterability methods.

Parameter	FilterKwik	SAST filterability
Sample mass (g)	180	225
Water grade and temperature	Heated household grade	Deionised at room temperature
Brix (%)	30	60
Temperature (°C)	70	20
pH	Natural	Buffered at 9.0
Filtration media	Glassfibre prefilter & 8.0 µm membrane	Whatman No 54 filter paper & Celite 505 filter aid
Pressure (kPa)	20.5	350

Buffering of the pH of sugar solution during the SAST filterability method is achieved using a calcium acetate solution. Since South African raw sugars tend to be high in phosphates, this buffer effectively initiates calcium phosphate precipitation which would artificially lower the filterability of a sugar. This was the main reason for choosing the use of membranes at a natural pH with FilterKwik (personal communication³).

In addition to the operational differences in Table 1, the FilterKwik procedure is simpler and more robust than the SAST procedure. It is also much quicker, except when poor filtering sugars are analysed in which case the analysis times are about the same. Moreover, the FilterKwik procedure allows for additional investigation into the causes of and contributing factors to poor filtering sugars through analysis of the residue left on the membrane after filtration.

Raw sugar starch, dextran, colour and pol

Starch levels were determined using the SMRI starch method (Anon, 2009a) and dextran levels were determined using the haze method (Anon, 2009b). Colour and pol values were determined according to official methods of ICUMSA

Results and Discussion

Repeatability limits

The repeatability limits of the FilterKwik method were determined on four different sample sets, viz:

1. Accreditation control A samples (May-September)
2. Accreditation control B samples (August)
3. Accreditation control C samples (September-November)
4. Two hundred and thirty-nine different raw sugar samples.

Results of the repeatability evaluations are shown in Table 2.

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Table 2. Basic statistics and repeatability values for FilterKwik.

Parameter	Control A	Control B	Control C	Sugars
Number of duplicate tests per sample	2	2	2	2
Number of outliers removed	0	1	1	0
Number of samples	37	10	27	239
Mean value (%)	50	52	65	n/a ^α
Repeatability standard deviation (s _r) (%)	4	4	10	n/a
Coefficient of variation (CV _r) (%)	9	8	16	n/a
Repeatability limit (r) (%)	3	3	4	3

^α Seasonal sugars with different filterability values

The repeatability limit for the FilterKwik method is therefore $\pm 2\%$ ($r=4\%$) which is equivalent to the tolerance used with the SAST method (Anon, 2002). Therefore, in all subsequent data evaluations, only the average FilterKwik duplicate values were considered.

Comparison of FilterKwik and the SAST filterability results

Direct correlations

Results of the FilterKwik and the SAST filterability methods are not expected to show any correlations since the two methods are fundamentally different (refer to Table 1 in Experimental). Lionnet (2002) found correlations between the SAST results and the filtration rates but not the final filtrate volumes.

Nonetheless, data evaluation was done to determine the level of agreement between the two methods with the main intention of facilitating a smooth transition from the SAST method to FilterKwik in the South African sugar industry. Datapoints of FilterKwik versus the SAST filterabilities are shown in Figure 1.

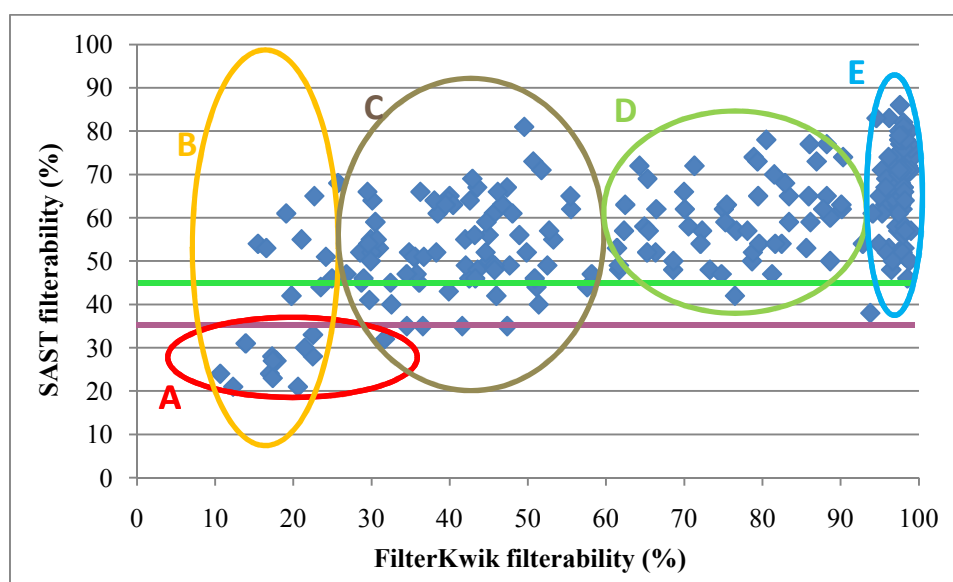


Figure 1. FilterKwik versus South African Sugar Terminal (SAST) filterability data.

The lower horizontal (**purple**) line (35% on the y-axis) indicates the current SAST filterability penalty level (<35%). The A-oval (**red**) indicates the sugars that were penalised for low filterability according to the SAST method. The upper horizontal (**green**) line (45% on the y-axis) indicates the current industry filterability standard ($\geq 45\%$). Using the FilterKwik data, the B-oval (**orange**) indicates the poor filtering sugars, the C-oval (**brown**) medium filtering sugars, the D-oval (**green**) the high filtering sugars, and the E-oval (**blue**) the very high filtering sugars. From the A-oval (**red**) all of the 2009/10 filterability penalty sugars except one, were successfully identified by the FilterKwik method. An additional eight samples were flagged as poor filtering sugars according to the FilterKwik results (<25%).

Through further interrogation of the data it became clear that there were inherent filterability differences between sugars from different factories. This was shown through vastly different trends when comparing the two methods per factory and confirms that historical data cannot be related to future FilterKwik data through the application of factors or equations. To elucidate these trends further, linear best fit correlations were collected for individual factories, as shown in Table 3.

Table 3. Linear best fit data for FilterKwik vs SAST filterability ($y=mx+c$).

Factory	m	c	Correlation coefficient (R)	Number of samples
All together	0.31	37	0.65	210
MS	0.15	57	0.40	5
AK	0.26	39	0.45	5
PG	-0.19	61	0.41	13
FX	0.03	51	0.08	16
KM	0.43	36	0.82	18
DL	0.41	25	0.83	18
UF	0.06	63	0.20	25
ES	0.34	41	0.88	27
UK	0.37	21	0.88	27
UCL	0.33	33	0.75	28
SZ	0.42	29	0.90	28

$y=mx+c$ is the best fit linear curve through the data using the method of least squares

Note that the slopes for the FX and UF data were approaching zero, while PG showed a negative correlation, albeit with a lower correlation coefficient. The only factories that showed good correlations were SZ, ES and UK; the linear best fit equations for data from these factories were noticeably different.

Ascending FilterKwik plots

When the FilterKwik data was plotted with the SAST data on the same y-axis but in increasing FilterKwik value order, there was, despite the wide scatter, a general increase in the SAST data (Figure 2).

The FilterKwik values varied from 10 to 100%, whereas the SAST values varied between 20 and 85%. FilterKwik is therefore sensitive to the nature of the actual sugars over a wider range and, more specifically, focussed on distinguishing between the lower filtering sugars, rather than the higher filtering sugars.

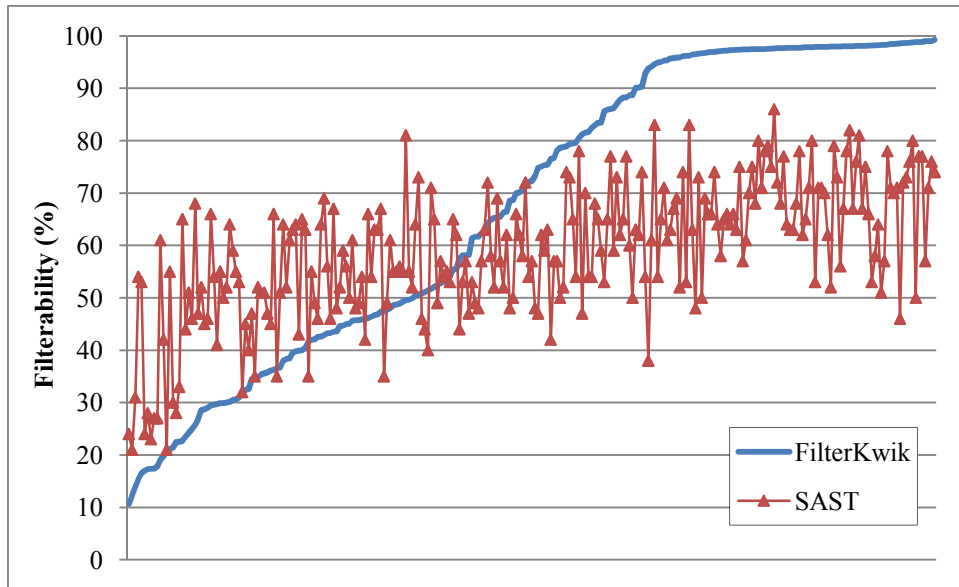


Figure 2. FilterKwik and SAST filterability in increasing FilterKwik value order.

The effect of some sugar quality parameters on filterability

The raw sugars were also analysed for starch, dextran, ICUMSA colour at pH 7 and pol. In Figures 3 and 4, the FilterKwik and SAST results were plotted on the same (y-) axis against starch or dextran, respectively, and each time in increasing order of either starch or dextran. Due to the large volume of data, such presentation was found to be most beneficial for relevant visual observations. Correlation graphs and linear fit equations did not offer any further insight.

Figure 3 shows the FilterKwik and SAST filterability values in order of increasing starch levels from 0 to 390 ppm. While there were no statistically significant trends, the filterabilities of both methods appeared to be affected by the starch levels with low starch levels up to 65 ppm exclusively coinciding with high filterabilities ($\geq 45\%$). On the high end of the starch scale, all of the sugars with starch levels higher than 140 ppm had SAST filterability levels of less than 57%, as indicated by the red oval. The SAST starch penalty level is currently 130 ppm. FilterKwik results did not show any effects coinciding with high starch levels.

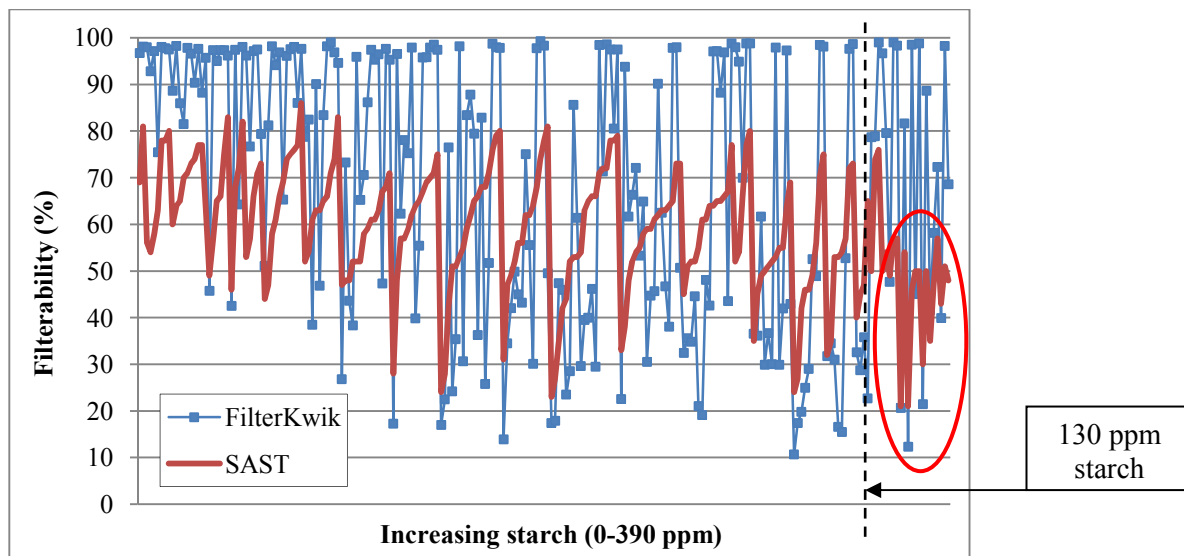


Figure 3. FilterKwik and SAST filterabilities plotted in increasing starch value order.

Figure 4 shows the FilterKwik and SAST filterability values in order of increasing dextran levels from 0 to 750 ppm. No significant trend could be established between dextran levels and either of the filterabilities. However, this time a clear effect was observed on both filterability results when dextran levels increased above 145 ppm, as indicated by the red oval. This level coincides exactly with the export specification level of 145 ppm for dextran in raw sugar above which the dextran is deemed to affect the refining process negatively (Morel du Boil and Wienese, 2002). Remarkably, all of the SAST filterability penalty sugars for the season (Figure 1, A-oval) showed dextran levels above this threshold.

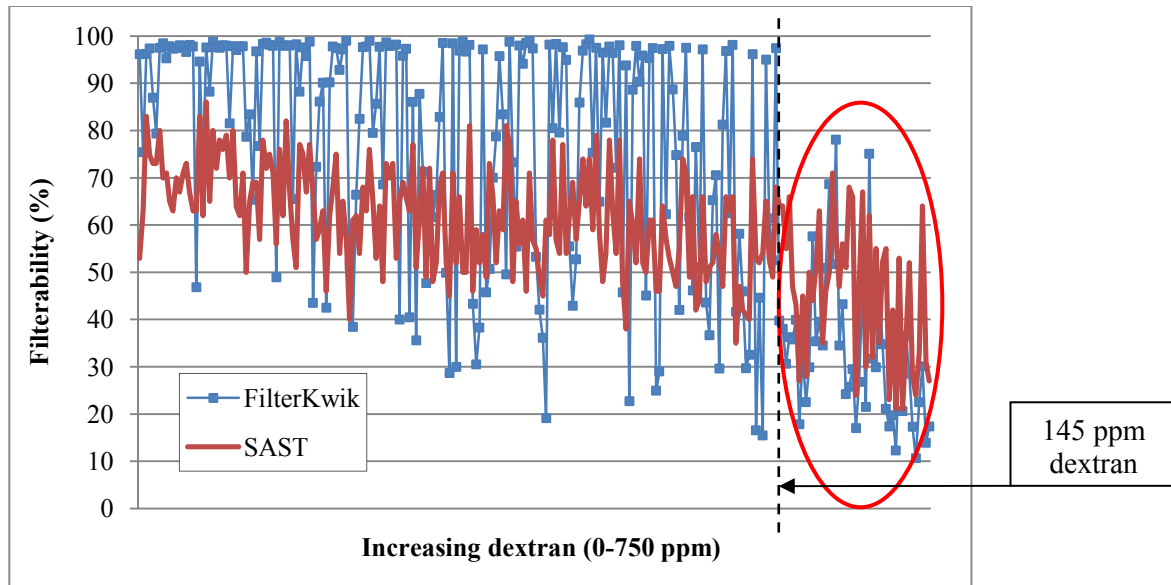


Figure 4. FilterKwik and SAST filterabilities plotted in increasing dextran value order.

While there appeared to be a mild trend between colour and filterability, with increased colour resulting in reduced filterabilities, this trend was not statistically significant ($p=0.05$). Similarly, an apparent trend between pol values and filterability with decreased pol values resulting in reduced filterabilities, was not statistically significant ($p=0.05$). The data does therefore not substantiate any contributing effect of colour or pol to the filtering quality of a raw sugar sample; there are, however, associations between colour values and filterability as well as pol values and filterability.

Residues left on the FilterKwik membranes

The FilterKwik procedure allows for additional investigation into the causes of and factors contributing to poor filtering sugars through analysis of the residue left on the membrane after filtration. Figure 5 shows pictures of membranes after filtration with powdery, grainy and bagacillo-rich residues, respectively, with 10x magnifications.

Residues can be subjected to individual elemental analyses and organic compound identification or general screening such as total inorganic and organic content to facilitate detection of the causes of poor filtration.

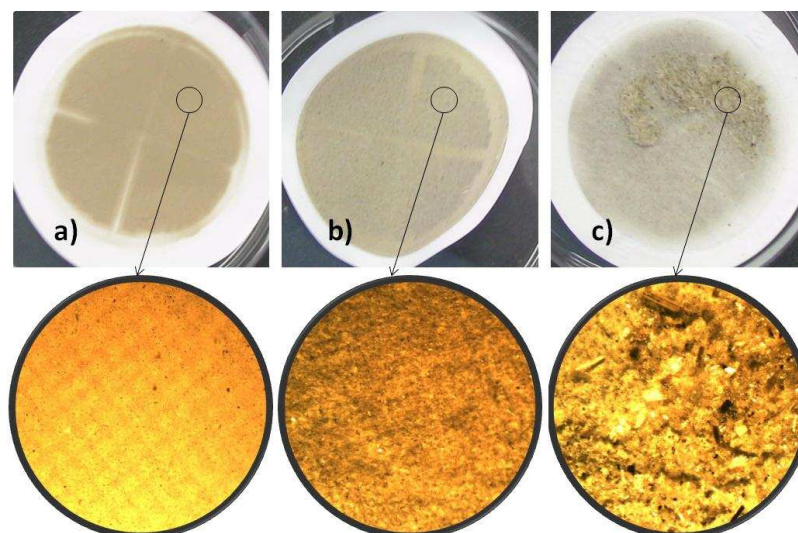


Figure 5. FilterKwik membranes after filtration with a) powdery, b) grainy and c) bagacillo-rich residues and a 10x magnification of each.

Conclusions

Since its inception, the FilterKwik apparatus has been subjected to rigorous testing, a series of standardisation and optimisation tests (unpublished), as well as a seasonal evaluation (2009/10) including comparisons to a range of other quality parameters. The repeatability limits of the procedure as determined during the seasonal evaluation are $\pm 2\%$. While there are no significant correlations with the SAST filterability results, FilterKwik can be used to predict the filtering behaviour of raw sugars destined for refining, be it local or abroad.

Evaluation of the data showed clearly that starch affects the SAST filterability at levels above 130 ppm. Both the FilterKwik and the SAST filterabilities were affected by dextran levels above 145 ppm, which is considered the threshold before refinery problems start to appear (Morel du Boil and Wienese, 2002).

The seasonal evaluation (2009/10) confirmed that the FilterKwik apparatus is fit for function.

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