REFEREED PAPER

ION EXCLUSION DESUGARISATION OF REFINERY JET 4 USING A SIMULATED MOVING BED PILOT PLANT

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Abstract

Ion exclusion is a chromatographic process which separates components from an impure feedstock on the basis of molecular size and charge. The idea of using ion exclusion to increase the recovery of sucrose from refinery Jet 4 (also known as return syrup, the run-off from the last white sugar boiling) has been considered for at least 20 years, but there are still no known installations using the technology for this application. The SMRI recently installed a simulated moving bed (SMB) ion exclusion pilot plant and used it to investigate the feasibility of desugarising refinery Jet 4. The results obtained are compared with those from a previous author who used a batch ion exclusion column to recover sucrose from Jet 4. Although not optimised in the current study, a sucrose recovery of 96% was achieved. The SMB was able to reduce the ash, colour and oligosaccharide components of Jet 4 by up to 93, 72 and 65%, respectively. Other methods of treating Jet 4 were also examined to understand under which conditions one technology is more suitable than another.

Keywords: ion exclusion, chromatography, desugarisation, simulated moving bed

Background and Literature Review

In this report, the term ion exclusion is used to describe both ion and size exclusion chromatographic separation. The principle on which ion exclusion is based is that larger molecules and charged ions will pass more quickly through a column, filled with the appropriate resin, than smaller or uncharged molecules. While ion exclusion is widely used to desugarise molasses in the beet industry, it has not yet become an established technology in the cane industry. Beet molasses contains less divalent ions, suspended solids and reducing sugars than cane molasses. Divalent ions reduce the efficiency of the ion exchange resin, and softening of the feed is therefore required. Suspended solids clog the resin bed, and must therefore be removed. Reducing sugars tend to remain with the sucrose in standard SMB desugarisation applications, and therefore reduce the purity of the sucrose-rich extract. Cane molasses contains about 15% reducing sugars compared with less than 1% for beet molasses. Modifications to the standard SMB ‘two product’ system, such as ARi’s ‘coupled loop’ process (Kearney, 1999) have the ability to remove reducing sugars. The following applications of ion exclusion in the cane industry have been previously examined.

Recovery of sugars as sucrose from raw house molasses

Thompson (1994) found the main challenge with using ion exclusion to desugarise raw house molasses is the pre-treatment of the molasses stream to remove divalent ions and suspended solids. In particular, both a chemical treatment and a physical separation were required to remove these impurities.

Recovery of sugars as invert from raw house molasses
Davis *et al.* (1997) used acidification to invert all the sugar in molasses, thereby enabling the sugars to be separated more effectively from the ash in the molasses. The suspended solids were removed by centrifugation before ion exclusion. The invert rich extract stream could be concentrated and sold as high test molasses (HTM) and the raffinate, with high levels of potassium, could be used as fertiliser. It appears that softening is not a requirement for the ion exclusion desugarisation of monosaccharides. Bernhardt *et al.* (1999) examined the economics of the process. At the time of the analysis, HTM sold for about five times the price of raw molasses, and about two tons of raw molasses were required to produce one ton of HTM. Stolz and Bekker (2001) developed a new pre-treatment process and then used an SMB to desugarise raw molasses. Recovery of sugar (as invert) ranged from 93.9 to 99.7% depending on the amount of water used.

Recovery of sucrose from clear juice
Kearney (1999) reported that clear juice purity increased from 86.2 to 97.0% through chromatographic separation. The colour of the extract was reduced from 18000 to 1300 ICUMSA[1] units. Sugar with a colour of 23 ICUMSA units could be crystallised from the evaporated juice

Purification of refinery affination syrup
Rein (2007) suggested applying ion exclusion to refinery affination syrup. The affination syrup would first require softening before chromatographic separation.

Recovery of sucrose from refinery Jet 4
Peacock (1996) used batch chromatography to show that it was possible to crystallise more sugar from Jet 4 by first removing some impurities using a single chromatography column. Prior to chromatography, the Jet 4 was chemically softened through sulphitation, defecation and filtration. The residual divalent ions result in a slow decline in column efficiency, requiring periodic regeneration of the resin beads with a concentrated brine of monovalent cations. The ion exclusion process increased the purity from 92.0 for Jet 4 to 97.9 for the ion exclusion extract. Kearney (1999) reported refinery molasses purity being increased from 84.3 to 97.1% through chromatographic separation. A colour elimination of 90% was also achieved.

Scope of the SMRI[2]'s 2014 Jet 4 work

In South Africa, Jet 4 is usually exhausted in a recovery house or recycled to a raw house. The disadvantages of long Jet 4 boiling times in the recovery house and reduced crushing capacity when recycling to the raw house have kept ion exclusion on the radar as a possible means for treating Jet 4. While Peacock's (1996) results showed the potential of ion exclusion for desugarising Jet 4, the disadvantages of the batch system were apparent, namely a high dilution of the Jet 4 and a large resin requirement. It was decided to use an SMB to desugarise Jet 4, and to compare the sucrose recovery, impurity removal, resin usage and water requirement with those of Peacock. A techno-economic analysis was also conducted in order to compare the economics of alternative Jet 4 treatment options with ion exclusion.

[1] International Commission for Uniform Methods of Sugar Analysis
[2] Sugar Milling Research Institute NPC

Equipment and Methods

Feed preparation

Jet 4 collected from a local refinery typically has a brix of 75%. It was diluted to 60% brix to reduce the pressure drop through the resin filled softening and ion exclusion columns. Before ion exclusion, the diluted Jet 4 was softened to reduce the divalent ion concentration to below 300 ppm as recommended by previous authors (Thompson *et al*, 1994). Softening was performed in batches by pumping 8 litres of Jet 4 through a glass column filled with 2.6 litres of ion exchange resin (Purolite® SST80DL). The resin was regenerated after each batch using 2 litres of a 10% brine solution. The Jet 4 normally required two passes through the column in order to reduce its cation concentration to below 300 ppm.

Batch test

The batch test was performed by pumping a slug of softened jet 4 through a fixed volume of resin, and following it with water (eluent). The composition of the outlet stream from the column was recorded as a function of the volume of liquid displaced from the column. Volumes recorded were expressed as unitless bed volumes (BVs) by dividing them by the bulk resin volume; this allows for comparison between plants of different size. Similarly, flow rates were expressed as BVs/h by dividing them by bulk resin volume. Six one litre columns (i.e. a bulk resin volume of six litres) from the SMB were connected in series for the 2014 batch test. The Jet 4 slug volume and eluent pumping rate were selected to be the same (on a BV basis) as Peacock (1996) used in his trials (Table 1).

Table 1. Batch test conditions for Peacock's (1996) trials and the SMRI 2014 trials.

	Peacock (1996)	2014 results
Resin volume (L)	200	6
Jet 4 slug volume (BVs)	0.005	0.005
Flow rate (BVs/h)	0.3	0.3

Pilot plant

A schematic diagram of the SMB pilot plant is shown in Figure 1. Eighteen columns, each containing one litre of resin (Purolite® PCR 642 in the sodium form), were connected to each other through a 'rotary multiport valve'. At regular time intervals the valve rotated (indexed) one position, thereby simulating the movement of resin counter-current to the direction of the juice flow. The shaded circles in Figure 1 represent the SMB columns; the more green the colour of the circle, the higher the sucrose purity; the lighter the shade of the circles, the lower the brix of juice in the column. The two inlet and two outlet streams divided the columns into four 'zones', namely, the enrichment, stripping, reload and elution zones. The high brix in the enrichment and stripping zones may result in high pressure drops through the columns. The columns in these zones were therefore each configured in parallel to reduce the pressure in the system. The point of highest pressure was located immediately after the enrichment zone pump and a pressure gauge at that point was used to ensure that the maximum working pressure (2 bar) of the plastic columns was not exceeded.

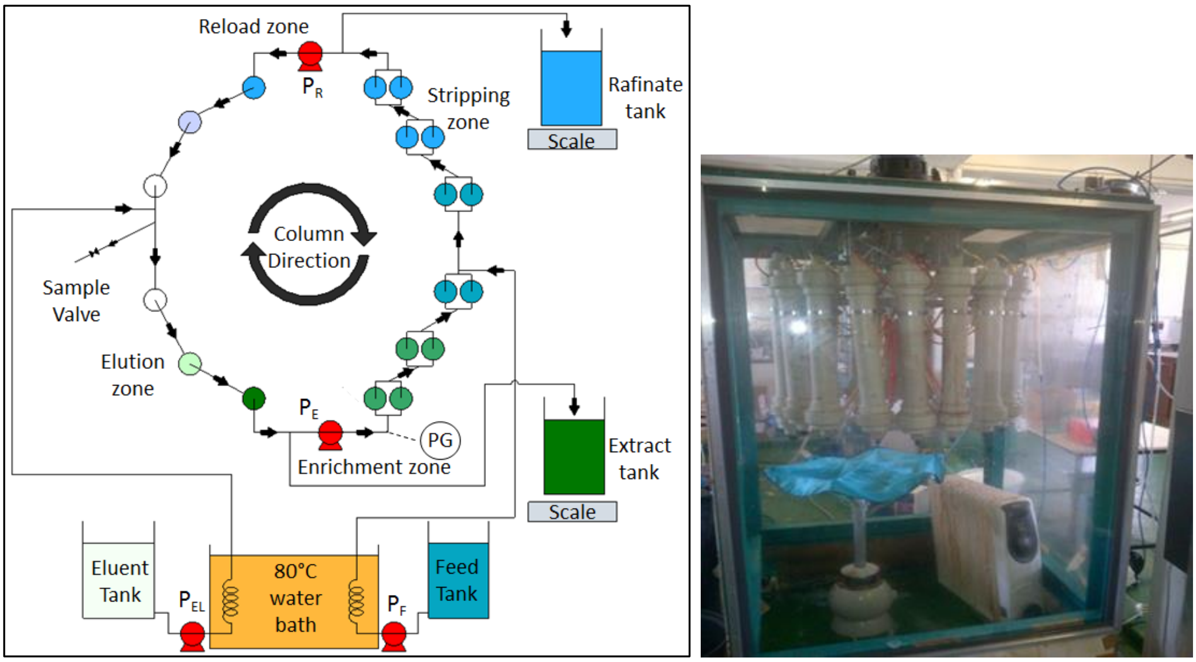

Figure 1. Schematic diagram and photograph of the SMB pilot plant used for the 2014 ion exclusion trials.

The entire SMB unit was placed within a heated enclosure maintained at about 50°C. The feed and eluent were heated before being pumped into the SMB. The higher temperature reduced the viscosity, thereby reducing the pressure drop through the columns.

Pilot plant operation

The flow rate in each of the four zones was adjusted to control how the constituents of Jet 4 were distributed between the two product streams. A sample valve was positioned as shown in Figure 1. The brix at this point should be as low as possible, indicating a minimal amount of separated product being recombined. The brix at the sampling point can be adjusted by changing the reload zone flow, the eluent flow, or the resin flow.

Results

Jet 4 composition

The composition of five samples of diluted Jet 4 after softening is shown in Table 2.

Table 2. Composition ranges of five samples of diluted Jet 4 after softening.

Brix %	55.6 - 60.0
Purity %	91.3 - 95.9
Oligosaccharides (ppm)	5396 - 8133
Conductivity ash (%)	1.3 - 1.5
Colour (ICUMSA units)	6250 - 6704
Monosaccharides (%)	0.9 - 3.0

Jet 4 and softened Jet 4 cation composition

The cation compositions of unsoftened and softened Jet 4 are shown in Table 3. It was usually necessary to pump the Jet 4 twice through the softening column in order to reduce the divalent ions to below 300 ppm.

Table 3. Cation composition ranges of softened and unsoftened Jet 4 diluted to 60% brix.

	Unsoftened	Softened (first pass)	Softened (second pass)
No. of samples	3	5	2
Na (ppm)	680 - 950	3770 - 5350	6985 - 7980
K (ppm)	1900 - 3050	160 - 980	100 - 170
Ca (ppm)	2495 - 4115	310 - 1385	130 - 260
Mg (ppm)	130 - 300	55 - 140	15 - 15

Batch ion exclusion test

The batch elution profiles are shown in Figure 2. Expressing displacement and flow rate in terms of BVs allows the 2014 results to be compared with those of Peacock (1996). The batch elution profiles indicate the degree of separation between different components that is likely to be achieved in the SMB. The more distinct the peaks, the easier it is to separate components from each other. Increasing the resin volume, or decreasing the slug volume, would improve the separation between the peaks. With an unlimited resin volume, complete separation between the various components is theoretically possible. The following components were measured in the product stream from the batch tests.

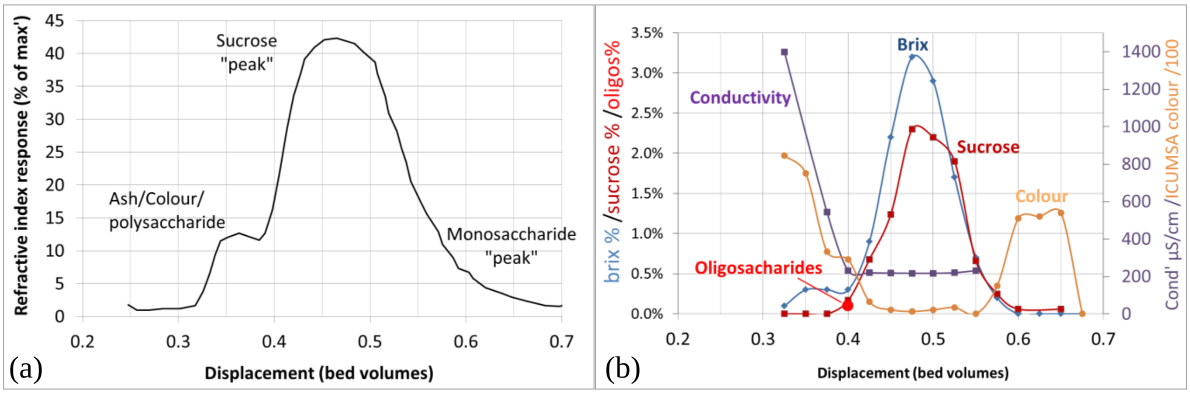

Figure 2. Batch test elution profiles for (a) Peacock's (1996) trials and (b) the SMRI 2014 trials.

Brix

The brix profiles for the 1996 and 2014 trials show a similar shape. In 2014, the eluted samples were analysed for conductivity, oligosaccharides, sucrose and colour and the results largely confirmed the 1996 prediction.

Ash

Charged particles are repelled by the charged surface of the resin bead, and thus do not flow into the pores of the bead itself. This results in charged molecules effectively having a shorter distance to travel through the column than molecules which are free to permeate through the pores of the beads. The ash peak occurs at about 0.35 BVs in both trials. For the 2014 batch trials, the relative ash content between samples was estimated by diluting all samples to 0.2% brix, and measuring their conductivities. Conductivities were therefore not measured for samples with brix readings below 0.2%.

Oligosaccharides

Oligosaccharides were eluted between the ash peak and the sucrose peak. Oligosaccharides consist of between 3 and 10 monosaccharide units, and thus it is expected they would elute before the disaccharide sucrose molecules which can more easily enter the resin pores.

Sucrose

It can be seen from the 2014 trials that the majority of the brix consists of sucrose, and the sucrose peak exits the column after the ash.

Monosaccharides

Monosaccharides are the smallest sugar molecules. It is therefore easier for them to enter the pores in the resin beads than the larger disaccharide and oligosaccharide molecules, and thus they are the last sugars to elute from the column. The exiting product from the 2014 batch column trials was not analysed for monosaccharides.

Colour

Colour molecules are generally large, complex and charged; they therefore are expected to exit the column before the sucrose peak. An interesting result from the 2014 trials, however, was the presence of a colour peak after the sucrose peak. Davis (2001) describes various types of colour bodies in cane sugar processing. Although usually charged, under certain pH conditions some colour bodies become neutral, which may allow them to enter the resin pores and elute after the sucrose peak. The juice pH was not measured during the trials, but varying this may change the effectiveness of the resin at removing colour. This result also suggests that Jet 4 contains some colour bodies that are smaller than sucrose.

Continuous ion exclusion test

The batch elution profile is useful for visualising and interpreting the SMB results. In a batch ion exclusion system where sucrose is the desired product, only the product eluted between 0.40 and 0.55 BVs (Figure 2) would be retained as extract. The early eluting and late eluting products are treated as waste. Reducing the product ‘area’ to between 0.45 and 0.5 BVs increases the purity of the extract, but decreases the recovery of sucrose. If a larger resin volume was used, then a more complete separation between the peaks could be achieved; in theory, both a 100% recovery of sucrose and a 100% purity extract are simultaneously achievable.

In a simple, two-product, SMB system the cut between the product and waste streams can only be made in one place. Given that most of the impurities in Jet 4 lie to the left of the sucrose peak, it is logical to make the cut between the ash ‘peak’ and the sucrose peak. The monosaccharides are therefore mostly included in the extract fraction. The exact position of the cut is a usually a compromise between the sucrose recovered to the extract, and the impurity removal (Figure 3). For a fixed resin flow rate, the position of the cut is determined by the liquid flow rate in the enrichment zone. A higher flow rate increases the removal of impurities to the left of the sucrose peak, while reducing the sucrose recovered to the extract. This can be interpreted in Figure 3 as shifting the ‘brix cut’ towards the right. While Figure 3 is useful for visualising the system, one of the advantages of an SMB is that the counter current flow simulates a long resin column; this improves the separation over a batch column by driving the sucrose and non-sucrose peaks apart from each other. Increasing the resin and internal juice flows further enhances this effect, but at the expense of a higher pressure in the system. The separation could also be enhanced by reducing the Jet 4 feed rate. The feed and resin flow rates were, however, kept constant for all the tests, with only the brix cuts being varied, and the results therefore do not represent the full separation capability of the SMB.

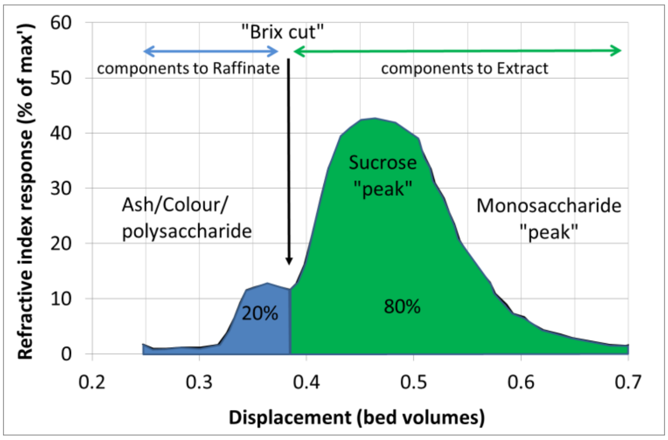

Figure 3. Illustration of an 80% "brix cut" in an un-optimised SMB, selected as a trade-off between impurity removal and sucrose recovery.

A number of runs were performed on the SMB with brix cuts varying from 79 to 96%. The results of component balance calculations (see Appendix), which estimate the removal of individual components at different brix splits, are shown in Figure 4. On a batch system, it is relatively simple to obtain the required brix cut; on an SMB, however, there is a possibility of separated products recombining, but fine tuning the system to avoid this happening was deemed beyond the scope of this study. The results should therefore be seen as a representation of general trends which were observed in an un-optimised system.

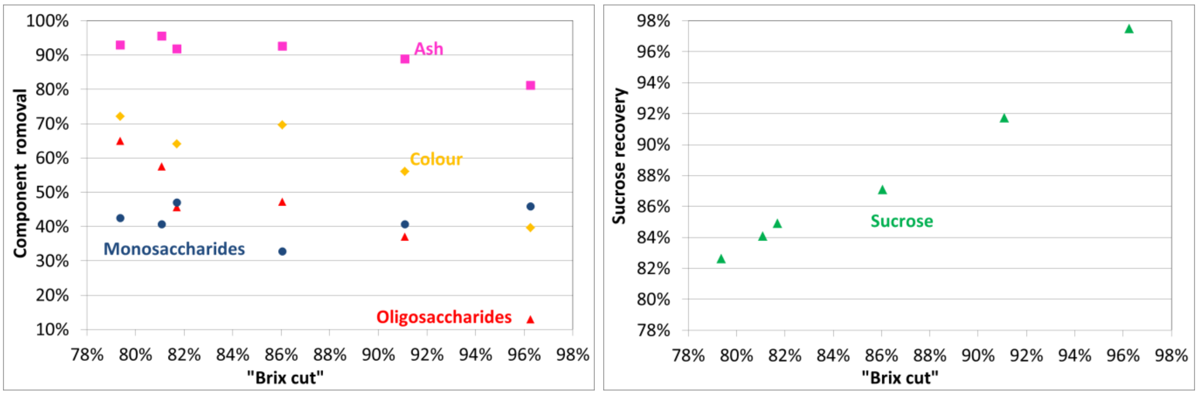

Figure 4. Mass balance results, showing the removal/recovery of various components of Jet 4 at different 'brix cuts'.

Comparison with Peacock's (1996) batch trials

Table 4 shows the conditions and results from the SMRI's SMB trials together with Peacock's (1996) batch column trials, allowing for a comparison between the two.

Table 4. Conditions and results of the SMRI's 2014 Jet 4 SMB compared with Peacock's (1996) batch ion exclusion results.

Conditions	SMB (2014)	Batch (1996)*
Feed rate (kg/h)	1.4	0.4
Resin volume (L)	18	200
Water/feed ratio	3	140
Resin flow (BV/h)	0.36	-
'Brix cut' (%)	79-96	72
Results		
Sucrose recovery (%)	83-97	76
Ash removal (%)	81-93	71
Monosaccharide removal (%)	33-47	31
Colour removal (%)	40-72	88
Oligosaccharide removal (%)	13-65	74

*Calculated from Peacock's (1996) data.

Resin volume and flowrate

Resin productivity, the amount of resin required to desugarise the Jet 4, is an important parameter required in the design of an ion exclusion plant. Resin productivity is often specified as the volume of resin required to process a certain volumetric flow rate of feed. For the batch column, the resin productivity was 650 m^3 resin/m^3/h feed versus about 16 m^3 resin/m^3/h feed for the SMB. No attempt was made to reduce this figure (by increasing the feed flow rate), but resin productivities of less than 10 m^3 resin/m^3/h feed are achievable on an industrial scale. Increasing the resin flow rate requires the internal juice flow rates to be correspondingly increased, and generally improves the separation. With a resin flow rate of 0.36 BV/h, the maximum pressure in the system was between 0.6 and 1 bar. The maximum allowable pressure in the columns was 2 bar, suggesting that the SMB could have been operated at higher resin and juice flow rates.

Dilution

One disadvantage with ion exclusion is the dilution of the feed with the eluent. With the batch column, the water to feed ratio is 140 versus 3 for the SMB. The result of this is a far more concentrated extract stream from the SMB which requires less evaporation. The extract brix from the SMB averaged about 25% versus 3% for the batch column.

Sucrose recovery and impurity removal

The sucrose recovery can be set to virtually any figure between 0 and 100%, depending on the brix cut. The batch system was operated at 76% sucrose recovery which should lead to a higher removal of impurities than the SMB. The results, however, show that the SMB was more effective in removing ash from the jet 4, even though the brix cut was higher. The counter-current nature of the SMB facilitates a better separation between the ash and sucrose than the static batch system.

The position of the monosaccharide peak after the sucrose peak in the batch profile (Figure 2) suggested that they would not be removed in the SMB. A monosaccharide removal of about 40% was, however, observed. It is expected that these monosaccharides passed around the 'back' of the SMB through the reload zone as the removal remained relatively constant, irrespective of the brix split. The sample valve brix varied between 1 and 3%. If the eluent and reload flows had been adjusted to reduce this brix further, it would have improved the recovery of sucrose, but reduced the monosaccharide removal.

While the batch column removed 88% of the colour, the SMB was somewhat less effective. The 2014 batch profile in Figure 2 suggests that a colour peak eluting after the sucrose peak may inhibit the effective removal of colour in a simple SMB. In a batch system, it is simple to discard the fraction which elutes after the sucrose peak, but at the expense of sucrose recovery. The oligosaccharide removal was at best 65%. The closeness of the oligosaccharide ‘peak’ to the sucrose peak (Figure 2) means it is difficult to remove oligosaccharides and maintain a high sucrose recovery. The lower sucrose recovery in the batch system may have been the reason why the oligosaccharide removal was higher there than in the SMB.

Alternative options for the treatment of Jet 4

SMB ion exclusion is one of many options available for gaining value from Jet 4. In order to compare the different options, a techno-economic analysis was performed (Jensen, 2013) based on a factory and refinery combination modelled using Sugars™.
The basis for the evaluation was:

- Crush rate: 300 tch
- Crush hours: 4200 hours per annum
- Steam/coal ratio: 8:1
- Coal price: R900/ton
- Raw (VHP) sugar price: R4200/ton
- Refined sugar price: R4950/ton
- Molasses price (85% brix): R484/ton.

For each of the options the capital cost of the main equipment was estimated and the annual revenue from the plant calculated. A rough comparison of which options recover the most sucrose as sugar can be seen by observing which options have the lowest purity reject (molasses or raffinate) streams. This trend is observed because the Jet 4 and sugar purity is roughly the same for all the options and an SJM recovery is therefore dependent only on the reject stream purity. A payback period for each option was calculated in order to provide a simple means of comparison. These figures are shown in Table 5. The options evaluated were:

1. Fermentation feedstock

The Jet 4 could be combined with raw house molasses and sold as a fermentation feedstock (at the molasses price) for the production of alcohol. This is considered the base case for the evaluation. The reject stream is Jet 4 at a purity of 93%.

2. Recovery house

The sucrose in Jet 4 could be further exhausted in a recovery house. The sucrose crystallised would be remelted with the raw sugar feed to the refinery. Long boiling times in the recovery house and recycling of impurities which tend to co-crystallise with the sucrose (oligosaccharides) are disadvantages of this option. The largest capital cost item was for a 25 m^3 continuous pan in the recovery house. The reject stream is recovery house molasses at a purity of 55%.

3. SMB

If an SMB is used for desugarising Jet 4, then the low brix, high purity extract stream could be returned to the remelter where the high water content could offset the amount of remelt water required. A sucrose recovery of 95% for the SMB separation was assumed; ash, colour, and monosaccharide removals of 97.6, 95 and 90% respectively were also assumed. The price of an SMB is highly variable, depending on the type, and the design fee charged by the manufacturer. Based on prices obtained, both low and high capital estimations were included in Table 5. At a conservative design resin productivity of 13 m^3 resin/m^3/h feed the SMB plant would require 30 m^3 of resin. The reject stream is evaporated raffinate at a purity of 40%.

4. Recovery house with recovery house molasses to SMB

The molasses leaving a recovery house still contains a substantial amount of sucrose (purity 55%). A small SMB could be used to recover the majority of this sucrose and recycle it to the refinery remelter. The advantage with this option is the much smaller SMB required (10 m^3 resin), and the ability to reduce the reject molasses from 40% sucrose purity to 11% with a sucrose recovery of 95% in the SMB. Once again, the analysis was performed for both low and high price estimations of the SMB. The reject stream is evaporated raffinate at a purity of 11%.

5. Recycling to a raw sugar factory

Where the refinery is in close proximity to the raw factory, Jet 4 can be recycled to the raw syrup tank. This is common practice with back-end refineries. This option, however, limits the flexibility for off-crop refining. There is still speculation as to whether the impurities in the recycled Jet 4 inhibit crystallisation in the raw house. A trial performed by Meadows and Sharma (2008) suggested that the effect is negligible, making this a very attractive option, especially in circumstances where excess raw house crystallisation capacity exists. For the analysis, it was assumed that an extra 21 m^3 of pan capacity in the raw house would be required, as well as an extra batch centrifuge. The reject stream is raw house molasses at a purity of 38%.

6. Treatment and sale as a speciality sugar

Given the high sugar content in Jet 4, it could be used as feedstock for high value speciality sugars. Further treatment might require de-ashing, inversion and decolourisation. Creating high value products from Jet 4 is certainly an attractive option, but given the variety of products which could be made, often using proprietary information, these options were not evaluated.

The results in Table 5 suggest that the most profitable option (which is also the simplest option) for treating Jet 4 is to recycle it to the raw house, and increase the raw house capacity if required. While boiling times in a recovery house are long, the cost of extra pan capacity is lower than the cost of an SMB. The option of using a small SMB to further desugarise the molasses from a recovery house provides the highest revenue, but it is capital intensive. For stand-alone refineries, without a market for speciality sugars, this might be the best option. A disadvantage of this option is the failure to remove the co-crystallising impurities in the Jet 4. It is recommended that the transfer and negative effect of these impurities in both the raw house and the refinery be further studied in order to assist with the decision as to which option is best, and under which circumstances.

Table 5. Summary of the techno-economic analysis results for various options for treating Jet 4.

Option	Increased revenue (R mil/yr)	Capital investment (R mil)	Payback period (months)
1. Fermentation feedstock (base)	0	0	0
2. Recovery house	29.70	9.0	3.6
3a. SMB (low estimate)	30.02	11.5	4.6
3b. SMB (high estimate)	30.02	37.0	14.8
4a. Recovery house with recovery house molasses to SMB (low estimate)	32.05	13.5	5.1
4b. Recovery house with recovery house molasses to SMB (high estimate)	32.05	22.0	8.2
5. Recycling to a sugar factory	30.97	4.5	1.7
6. Treatment and sale as a speciality sugar	?	?	?

Conclusions

Ion exclusion has yet to find its place in the sugarcane industry. Given its widespread usage in the beet and sweetener industry, it is expected that it will have a role to play in the cane based bio-refinery of the future. The SMRI installed and used an SMB pilot plant to remove impurities from Jet 4, and compare the results achieved with those from a previous author who used batch ion exclusion for the same purpose. The main improvement of the SMB over the batch system was the reduced water usage and resin requirement. Under the operating conditions tested, a trade-off existed between the removal of impurities and the recovery of sucrose. A sucrose recovery of 96% was achieved. The SMB was able to reduce the ash, colour and oligosaccharide components of Jet 4 by up to 93, 72 and 65%, respectively. As predicted from the batch tests, the SMB was less effective at removing monosaccharides (33 to 47% removal), and a more complex system would be required for this purpose. A techno-economic analysis was performed, with six Jet 4 treatment options being considered. The results suggest that using an SMB to desugarise molasses from a recovery house might be a better option than using it for Jet 4. Recycling Jet 4 to the raw house is the simplest and most profitable conventional option, assuming that the impurities in the Jet 4 do not negatively affect the crystallisation characteristics in the raw house. It is also worth seeking market opportunities for Jet 4, or treated Jet 4, as a speciality liquid sugar.

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REFERENCES

Anon (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition, Published by South African Sugar Technologists' Association, Mount Edgecombe, South Africa.

Bernhardt HW, Davis SB, Peacock SD and Walford SN (1999). Review of the process to make invert sugar from cane molasses *Proc S Afr Sug Technol Ass* 73: 235-240.

Davis SB (2001). The chemistry of colour removal: A processing perspective. *Proc S Afr Sug Technol Ass* 75: 328-336.

Davis SB, Peacock SD and Walford SN (1997). Ion exclusion chromatography: Molasses pretreatment and high test molasses production *Proc S Afr Sug Technol Ass* 71: 146-152.

Jensen PS (2013). Techno-economic analysis of potential refinery feedstocks for SMB desugarisation. SMRI Technical Note no. 09/13: 1-5.

Kearney M (1999). Advances in the ARi coupled loop chromatographic separation process *Proc of the Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, SPRI, Inc*: pp 163-172.

Meadows DM and Sharma P (2008). Exhaustion limits of refinery molasses analysis: Prediction, boiling-down tests and impact on raw mill exhaustion. *Tech Papers and Proc Ann Meeting Sug Ind Technol* 67: 116-136.

Peacock SD (1996). Ion exclusion desugarisation of refinery jet 4 *Proc S Afr Sug Technol Ass* 70: 171-176.

Rein PW (2007). *Cane Sugar Engineering*. Verlag Dr Albert Bartens. 529 pp.

Stolz HNP and Bekker PI (2001). Enhancement of sugar production part 2: Chromatographic separation of sugarcane molasses. *Proc S Afr Sug Technol Ass* 75: 351-357.

Thompson MC (1994). The potential application of ion exclusion chromatography for additional sucrose recovery from molasses. *Proc S Afr Sug Technol Ass* 68: 105-108.

Thompson MC, Morel du Boil PG and Moodley M (1994). Preliminary report on the recovery of sucrose from Jet 4 using ion exclusion chromatography. SMRI Technical Report no. 1688: pp 1-2.

APPENDIX
SMB mass balance calculations

The extract and raffinate streams from the SMB were directed into two separate tanks, located on two separate scales. The raffinate mass flow (M_{raf}) and extract mass flow (M_{ext}) were calculated by the change in mass on these scales. As the feed and eluent flows were not weighed, they were deduced from a mass balance and brix balance based on the outlet flows. The removal and recovery of individual components in Jet 4 were calculated as follows:

Brix
Brix was measured using a refractometer. Refractometer brix more accurately estimates dissolved solids concentration at higher sucrose purities than at lower sucrose purities. The proportion of brix exiting the SMB in the extract is calculated as:

$$\text{Brix recovery ('brix cut')} = M_{ext}*Brix_{ext}/(M_{ext}*Brix_{ext} + M_{raf}*Brix_{raf})$$

Sucrose
Sucrose was measured using HPLC. The proportion of sucrose exiting the SMB in the extract is calculated as:

$$\text{Sucrose recovery} = M_{ext}*Sucrose_{ext}/(M_{ext}*Sucrose_{ext} + M_{raf}*Sucrose_{raf})$$

Colour
Colour was measured by the ICUMSA method (Anon, 1985). A colour 'mass balance' was then performed, and the proportion of colour exiting the SMB in the extract is calculated as:

$$\text{Colour removal} = M_{raf}* Brix_{raf} *Colour_{raf}/(M_{ext}* Brix_{ext} *Colour_{ext} + M_{raf}* Brix_{raf} *Colour_{raf})$$

Ash
Conductivity ash measured in sugar solutions (Anon, 1985) assumes that the main cation present is potassium. The main cation present in softened Jet 4 is, however, sodium; this means that the conductivity ash method is likely to be inaccurate. The relative distribution of ash between samples was estimated by measuring the conductivity of all samples diluted to the same brix of 0.5% (Cond_0.5). This method assumes that conductivity varies linearly with ash concentration over the range measured. If not diluted to the same brix, then the conductivity measurement is mostly a function of how much water is present in the sample. What is required, however, is to compare the relative ash to brix ratios between samples, and measuring the conductivity of the samples diluted to the same brix allows this comparison.

$$\text{Ash removal} = M_{raf}*Brix_{raf}*Cond\_0.5_{raf}/(M_{ext}*Brix_{ext}*Cond\_0.5_{ext}+M_{raf}*Brix_{raf}*Cond\_0.5_{raf})$$

Monosaccharides (glucose + fructose)
Glucose and fructose were measured using an HPLC. Their concentrations were added together and the proportion of monosaccharides (Mono) exiting the SMB in the raffinate is calculated as:

$$\text{Monosaccharide removal} = M_{raf}*Mono_{raf}/(M_{ext}*Mono_{ext} + M_{raf}*Mono_{raf})$$

Oligosaccharides
Oligosaccharides were measured using an HPLC. The proportion of oligosaccharides (Oligo) exiting the SMB in the raffinate is calculated as:

$$\text{Oligosaccharide removal} = M_{raf}*Oligo_{raf}/(M_{ext}*Oligo_{ext} + M_{raf}*Oligo_{raf})$$