AN OLD TECHNOLOGIST'S PERSPECTIVES OF NEW SEPARATION TECHNOLOGIES

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Introduction

Chromatographic techniques, membrane filtration processes, softening, and electrodialysis have been attracting the attention of sugar researchers for a number of years; these concepts are seen here as falling under the "new separation technology" heading. There is no doubt that they are being used in the beet industry but their industrial application with cane has not been very successful. There is however a large amount of work which is being done to investigate the impact, particularly as far as recovery and sugar quality are concerned, of these techniques in the cane industry.

The approach taken here has been to concentrate on the broad findings of the experimental work with these new technologies, as applied to the cane industry. These findings have then been used to construct a number of operational schemes, relevant to the South African industry, where one or more of the new technologies is introduced. It is impossible to produce detailed material balances and performance data, but broad outlines can be given for the schemes which are seen as technically achievable.

There are serious limitations in the present approach. Financial aspects have been totally ignored, this exercise being seen as purely technical. The energy implications have also been ignored, except that additional evaporation or heating is mentioned. The treatment or disposal of the separated impurities, of washings, of regenerants, etc. has not been considered, except through attempts at estimating undetermined losses.

The present work is seen only as an attempt to look at new technologies and to propose, in principle, applications which are industrially achievable and relevant to the local industry.

Schemes with New Technologies

Base case

A typical South African situation will be used. The base case starts with 100 tons per hour of a normal South African mixed juice which is then clarified by the usual hot liming (milk of lime) technique used in the majority of South African factories. The qualities of the mixed and clear juices are representative of normal operations.

The clear juice is then evaporated to syrup, with colour increasing by about 3%.

The pan house is again typical of South African operations. Three massecuites are boiled, all the C-sugar is remelted; some B-sugar goes to magma for A-massecuite and the rest is remelted. VHP (very high pol) sugar is produced.

Under normal conditions South African factories report a sucrose undetermined loss of 2%. This has been distributed as 1% before syrup and 1% after syrup, based on balances involving syrup weighing.

Exhaustions and sucrose lost to final molasses are also typical. The final molasses purity has been taken as target purity, namely,

$$\left\{43.1 - 17.5\left(1 - e^{-0.74\frac{F+G}{A}}\right)\right\}$$

plus 2 units, to represent a realistic performance.

The material balance, which involves brix, pol, sucrose and colour is shown as Material Balance: BASE CASE.

We will now consider three scenarios, based on information available from the relevant literature.

Case 2: Membrane filtration of clear juice

In case 2, the clear juice is subjected to membrane separation processes. We now use the data found in the literature to obtain relevant results for the treated clear juice. The following has been assumed for the balances:

- A small increase in purity from 86.0 to 86.5.
- A 10% decrease in colour, from 20 000 to 18 000.
- A retentate mass equal to 10% of the feed mass but containing only 1.2% of the brix and 0.7% of the pol.
- A 75% removal of gums.
- A 99% removal of suspended solids.
- No change in the concentrations of sulphated ash, fructose, glucose and brix.
- An increase of 0.15% in undetermined loss.

No other effects have been considered, and the balance over the boiling house proceeds normally.

Clear juice has been used as the original material because there are strong indications in the literature that cane mixed juice cannot be handled successfully by the membranes presently available.

Case 3: Membrane filtration plus softening of clear juice

The permeate from the treated clear juice is now softened. Again the following has been assumed:

Inorganic Species	mg/kg Juice	
	From	To
Calcium	200	30
Magnesium	100	15
Sodium	10	1000
Potassium	1000	10

The impact of the concentration changes of Ca^{2+}, Mg^{2+}, Na^{+} and K^{+} has however been taken into consideration, using Sahadeo's (1998) results concerning the relevant melassigenic effects of these ions. The sulphated ash level has thus changed from 0.5% to 0.84% to quantify the new melassigenic effect. A new target purity has then been calculated for the final molasses and 2 units added to it.

- Undetermined loss has been increased by 0.15%.

Case 4: Membrane filtration plus softening of clear juice, followed by chromatographic separation

The syrup produced in Case 3 is now sent to a chromatographic separation process, to produce an "extract" containing about 78% of the brix, 89% of the pol (98 purity), and about 10% of the colour.

The "invert" stream contains most of the impurities.

The "extract" is now used as is, to produce refined sugar, through a conventional South African back-end refinery process. The following assumptions have been made:

- The colour transfers have been decreased from the usual values (0.04, 0.032 and 0.024) to 0.007 for all three boilings, on the strength of published experimental data from the pilot crystallisation of chromatographic extracts, mostly in the USA. These low transfer values may be too optimistic under South African conditions.
- The refined sugar colour is low at 28. This depends very much on the colour transfers used. A material balance for a conventional back-end refinery (carbonatation plus sulphitation) for similar materials and colours gives the same amount of refined sugar but at a much higher (152 instead of 28) colour.

  The extract colour (2200 units) is much higher than that of conventional South African refinery fine liquors (300 - 700 units); this range of fine liquor colours is required to yield refined sugar colours of 30 to 45 units, with conventional colour transfer values.

  The success of the scheme shown here with the chromatographic extract thus depends entirely on the colour transfers obtained with this type of material.
- Undetermined loss has been kept at 1.30% from mixed juice to syrup and 0.6% has been taken for the crystallisation, a typical value for a back-end refinery.
- The third run-off has not been recirculated back to the raw house. We have simply assumed an SJM recovery for this stream.
- The extract is at a relatively low brix (40) and needs to be concentrated to the usual pan liquor feed brix (about 72).

Selected results from the various schemes are summarised in Table 1.

The results in Table 1 do not include all the benefits of the various operations. Lower viscosities, better sugar quality, faster crystallisation rates, etc., are expected.

Cases 2, 3 and 4 provide interesting results and indicate the performances that can be achieved through the new technologies. It is felt however that the introduction of these technologies, as described here, in the South African industry will not be considered in the near future. Thoughts must be given to processes which are more specific to local conditions and which would replace, rather than add, unit operations.

Case 5: Pretreatment in the diffuser, followed by new separation technologies, resulting in the elimination of classical clarification

Most if not all of the applications of membrane technology reported in the cane literature (Kwok, 1996, Kochergin *et al.*, 2000; Kochergin, 1999; Eringis and Eaton, 2000; Wittwer 1999; Steindle and Doyle, 1999; Saska *et al.*, 1999) show that only clear juice (i.e. not mixed juice) can be treated by the membranes which are currently available. This indicates clearly that some form of pretreatment of the raw cane juice is essential. The classical raw cane juice treatment has always been through the use of heat and of lime.

A cane diffuser lends itself well to this pretreatment. In South Africa, diffusers are operated at 80 to 85°C. Barker and Lionnet (2000) show that the use of saccharate can facilitate liming in diffusers. Finally, Rein (1995) and Koster (1995) show that diffuser juice contains much less suspended solids (0.2%) than mill juice (0.6%). In 1998, Meadows showed that it is possible to route clarifier muds directly back to the diffuser. This is now done by five South African factories. These observations show that it should be possible to produce a treated diffuser juice which would be suitable as a feed for plants based on the new separation technologies. One aspect needing attention is liming. In normal mill juice clarification, liming has a dual role; it corrects the pH to control inversion but also causes the removal of suspended matter through flocculation and settling. With diffusion much suspended matter is removed by the bagasse bed. Liming is thus needed mostly to control pH. Schäffler *et al* (1985) have a model to estimate inversion losses as a function of pH, temperature, brix and time. This was used to estimate pH effects on the inversion of sucrose in diffuser juice at 85°C; the results are in Figure 1. It is clear that, at a pH of 6.4 to 6.6 inversion losses would be about 0.025%.

Liming tests were carried out to estimate the volume of saccharate needed to correct the diffuser juice pH. Catch samples of juice were taken and saccharate added; the volume of saccharate and the pH values were noted. For a pH of 6.6 and at 100 TCH, about 1200 litres of saccharate per hour would be needed. This saccharate would be prepared with un-weighed diffuser juice.

The treated juice would then have a composition close to the average of South African clear juice. If necessary it could be

Table 1. Selected results from the various operations.

	Sample	Purity	Colour	$\frac{F+G}{A}$	Suspended solids (%)	Tons	BHR	Undetermined loss (%)	Tons sucrose lost in final molasses
Base Case	MJ	86.0	20000	1.11	0.10				
	CJ	86.0	20000	1.00	0.05				
	Syrup	86.0	20600	-	-			2.00	0.93
	A-sugar	99.6	1236	-	-	9.31	88.0		
	Final molasses	35.95	-	-	-				
Case 2	MJ	86.0	20000	1.11	0.10				
	CJ	86.5	18000	1.00	0				
	Syrup	86.5	18540	-	-			2.15	0.88
	A-sugar	99.6	1112	-	-	9.22	88.0		
	Final molasses	35.95	-	-	-				
Case 3	MJ	86.0	20000	1.11	0.10				
	CJ	86.5	18000	0.59	0			2.30	1.02
	Syrup	86.5	18540	-	-				
	A-sugar	99.6	1113	-	-	9.15	87.4		
	Final molasses	38.88	-	-	-				
Case 4	MJ	86.0	20000	1.11	0.10				
	CJ	86.5	18000	0.59	0				
	Syrup	98.0	2110	-	-			1.95	
	Refined sugar	99.96	28	-	-	8.01	-		
	Jet	84.25	18826	-	-				0.97

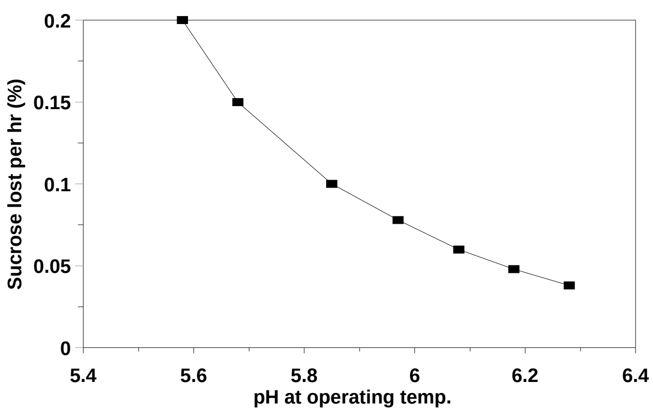

Figure 1. The effect of pH on the inversion of sucrose at 85°C.

re-heated to 85°C in juice heaters. It would be screened, pre-filtered and then sent to new separation systems. The advantages, shown in Case 2 would be reached but without the need for a flash tank, clarifiers and mud handling; classical flocculants would not be needed but other chemicals would be required for the separation plants.

Recent work at the SMRI has dealt with the laboratory clarification of cane juice under reproducible conditions. Some aspects of the composition of mixed juice and that of the corresponding clear juice are shown in Table 2.

The most noticeable changes are with pH, inorganic phosphate and silica. Suspended solids were not measured, but data are available, as discussed earlier. The results in Table 2 indicate that a pretreatment in the diffuser would produce a juice whose composition would not be vastly different to that of clear juice. A further correction for pH may be necessary, to ensure the desired downstream pH values; at this stage it is felt that this correction could be done with sodium bicarbonate or carbonate.

Table 2. Composition of MJ and of the corresponding CJ, from laboratory clarification.

	MJ	CJ
pH	5.1	7.0
Sulphated ash (%)	0.40	0.43
Inorganic phosphate (mg/kg brix)	1920	300
Calcium (mg/kg brix)	2000	2600
Magnesium (mg/kg brix)	1730	1540
Potassium (mg/kg brix)	8930	8880
Silica (mg/kg brix)	670	350
Purity (pol/brix)	85.0	86.3

Acknowledgements

The author is very grateful for the help received in preparing this paper, and would like to acknowledge inputs from the followings:

Brian Barker (SMRI) who carried out the saccharate liming experiments; Brian Purchase (SMRI) for useful discussions; Ken Koster (Illovo Sugar) for discussions on pH correction; Kevin Schäffler (SMRI) for the calculations of sucrose inversion; Lola Naidoo (SMRI) for all the drawing and visuals, and for extreme patience towards the author; Prem Sahadeo (SMRI) for all the material balances; and Geneviève Thélémaque (SMRI) for typing a difficult manuscript.

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