A COMPARATIVE STUDY OF SUCROSE DEGRADATION IN DIFFERENT EVAPORATORS

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Abstract

Theoretical calculations based on measured conditions of pH, brix, temperature and residence time suggested that sucrose degradation in the Pongola evaporator was almost 0,5% and that almost all of the loss occurred in the first two effects of the evaporator. When data from all South African factories were considered there appeared to be a correlation between the relative size of the first two evaporator effects and the undetermined loss. Sucrose degradation in 5 different evaporators was then monitored by measuring changes in glucose:sucrose ratios.

Changes in juice pH, and concentration of acetic acid in condensates were also monitored. These measurements have indicated that appreciable degradation occurs in two of the evaporators (Pongola and Felixton). At both of these factories a large portion of the evaporation is done in the first two effects whereas at Mount Edgecombe where much less evaporation occurs in the first two effects no such degradation could be detected. The measured inversion for all five evaporators correlates well with the area of heating surface in the first two effects per unit of sucrose throughput. The observations illustrate that energy savings made by extensive vapour bleeding may sometimes be worth less than the consequential destruction of sucrose.

Introduction

Investigations of undetermined losses at Pongola (PG) led to a suspicion that sucrose degradation was occurring in the evaporator[3]. This initiated a detailed study of the evaporator followed by a comparative study involving four other evaporators.

The measurement of low levels of inversion in evaporators is difficult. It generally cannot be based on sucrose mass balances because of the limited precision of the weighing and analyses. Low levels of glucose in juices can be measured more precisely than the relatively large quantities of sucrose and so sucrose destruction is best estimated indirectly from changes in glucose:Bx or glucose:sucrose ratios (Schäffler *et al.*[4]). This paper summarises the results of more than 600 such analyses based on more than 1 200 samples collected from evaporators.

Methods

Theoretical calculations of inversion

Vukov's[6] formula was used for estimating sucrose inversion in different evaporator vessels. To apply the formula it is necessary to know (a) the juice temperature and brix, (b) the juice pH at the operating temperature and (c) the residence time in the vessel.

At PG temperatures were measured by using thermocouples placed in oil-filled pockets in the juice exit pipes. To determine the pH at operating temperature several juices from various stages of the evaporator were heated slowly to 85°C whilst the pH was recorded (the pH meter was calibrated at different temperatures with buffers of known temperature sensitivity). A relationship between pH at 22°C and change in pH per unit change in temperature was then established and used to estimate pH at operating temperature[5].

Residence times were calculated from juice flow rates and vessel volumes, the latter being calculated from dimensions given in engineering drawings. It was generally assumed that the juice height in tubes was 30 cm. For Kestners the residence time calculations were based on the inlet juice flow rate; for the 1C vessel (semi-Kestner) it was based on the mean of the inlet and outlet flow rate, and for all other vessels it was based on the outlet rate. As a check on the calculated residence times, tracer tests were carried out across various stages of the PG evaporator. Good agreement was obtained for vessel 1C but the calculated residence times for vessels 2A and 2B combined were shorter than those actually measured (calculated for 250 t h^{-1} clear juice = 11 min.; measured = 14 min.) so the theoretical estimates of inversion in the second effect are probably conservative.

Measurements of inversion

Gas chromatography (GC) was used to measure changes in glucose:sucrose ratios across evaporators. For comparisons between evaporators, samples were taken by factory staff once per shift throughout one week of each month of the 1986/87 season. The streams sampled were (i) clear juice, (ii) juice exiting the second effect and (iii) syrup. Condensates of vapours 1, 2 and 3 were also sampled for acetic acid analyses.

At PG the 1C vessel was by-passed for almost the entire 1986/87 season and all sets of samples collected by factory staff were taken whilst the vessel was by-passed. For investigations of the 1C vessel the sampling procedure was more sophisticated in that a calculated time delay was built in with the intention of ensuring that each set of downstream samples was derived from the same parent juice. The samples were collected by Sugar Milling Research Institute (SMRI) staff over a single day. A similar sampling procedure was applied by SMRI staff at Felixton (FX) when comparing different flowrates through the separate evaporator sets.

All the juice samples were analysed for glucose, fructose and sucrose (GC), brix (refractometer) and pH (at ambient temperature after dilution to the brix of the associated clear juice sample). Vapour condensates were analysed for acetic acid by GC.

Results and Discussion

Pongola

Numerous brix and pH profiles were measured across the PG evaporator and used to calculate inversion. Some pH results and a sample spreadsheet calculation are shown in the Appendices. The mean results from 32 such calculations are shown in Table 1.

Theoretical calculations of inversion cannot be highly accurate because:-

• For vessels in the first two effects there is an appreciable difference in juice pH between the inlet and outlet samples

Table 1

Mean results from 32 theoretical estimates of sucrose inversion in the Pongola evaporator

Vessel(s)	Kest's	lc	2a+b	3a+b	4a+b	5a+b
Inversion (%)	0,04	0,23	0,15	0,04	0,01	0,00
Total (%) Range (%)	0,47 0,19–0,68					

(Appendix 1) and therefore it is not possible to determine the effective operating pH in the actual vessel. For the calculations summarised in Table 1 the inlet pH has been used and therefore inversion estimates must be conservative.

- In calculating residence times, assumptions are made regarding volumetric flow rates.

Despite their limited accuracy the theoretical estimates provided a useful indication of the extent and location of sucrose degradation. In particular these estimates indicated that appreciable losses were occurring (0.5%) and that almost 90% of the losses took place in the first two effects of the evaporator.

The theoretical indication of high inversion losses is supported by results obtained with syrup weighing and analyses during the preceding 4 seasons. Mass balances showed a fairly consistent loss of sucrose between mixed juice and syrup but a gain in glucose. Changes in glucose:sucrose ratios between mixed juice and syrup regularly indicated sucrose losses of about 0,5%[3].

The observation that almost 90% of the inversion in the PG evaporator took place in the first two effects prompted a consideration of other evaporators in the industry. Data on the distribution of heating surfaces in each evaporator were obtained from Allan[1]. An apparent relationship between distribution of heating surfaces and undetermined loss is shown in Figure 1. This relationship reinforced the suspicion that part of the undetermined loss problem at PG is caused by the front end of the evaporator.

Other Evaporators

The investigation of inversion in evaporators was continued by monitoring four evaporators throughout the 1986/87 season. They were chosen to represent a range of heating surface distributions. Facilities for by-passing the 1C vessel at PG were installed prior to the 1986/87 season so it became possible to operate the evaporator with different amounts of first effect heating surface.

Results of measured changes in glucose % sucrose across the different evaporators are shown in Table 2. They confirmed that when appreciable inversion occurred it was mostly within the first two effects and that the highest rates of inversion were generally associated with evaporators in which a relatively large portion of heating surface was located in the first two effects. The figure for PG with the 1C vessel operating was obtained when there was a high throughput of 255 to 260 t h⁻¹ of mixed juice. It agrees well with that measured throughout the 1984/85 season between mixed juice and syrup. In previous seasons, when the mixed juice throughput was lower, the increase in glucose % sucrose was frequently above 0,25% and seldom below 0,15%[3].

Measured changes in glucose % sucrose cannot give an accurate assessment of sucrose degradation because an unknown amount of glucose is destroyed during evaporation (hence some negative figures for Mount Edgecombe (ME).
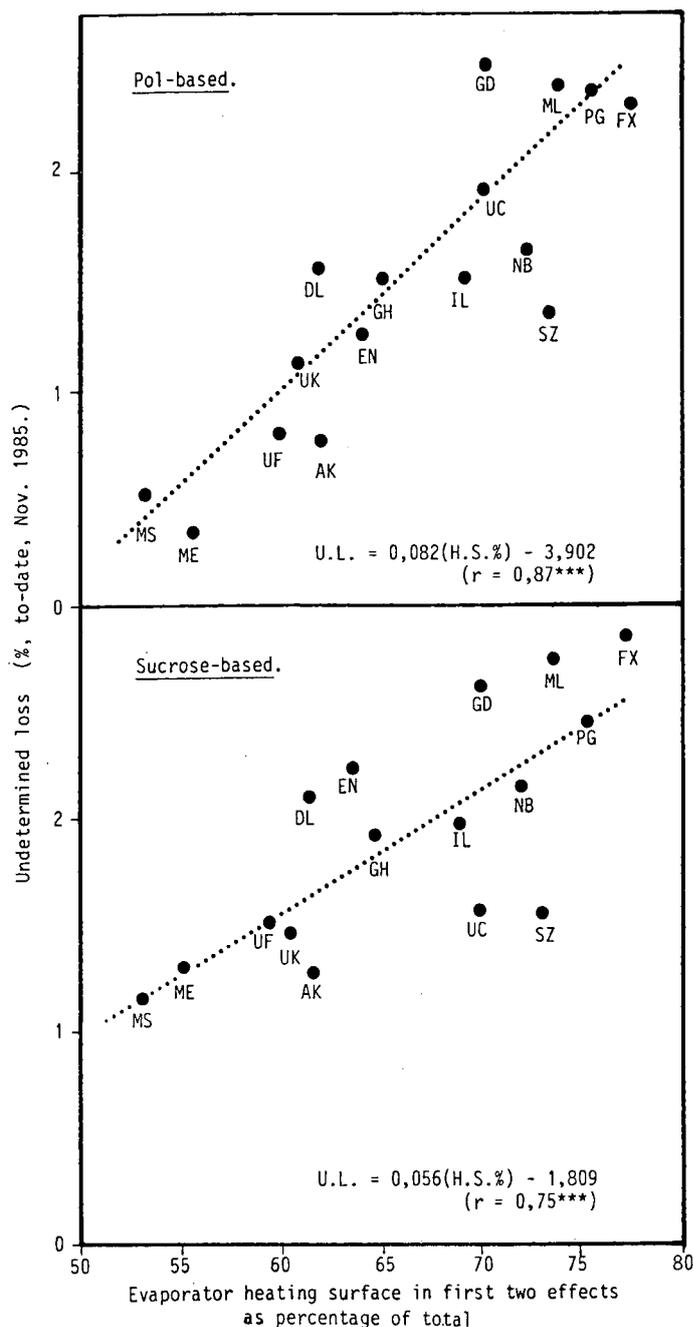

U.L. = 0,082(H.S.%) − 3,902
(r = 0,87***)

U.L. = 0,056(H.S.%) − 1,809
(r = 0,75***)

FIGURE 1 Undetermined losses at South African sugar factories, and the portion of evaporator heating surface in the first two effects.

They can however indicate the minimum sucrose degradation, which is approximately 1,9 times the change in glucose % sucrose. Thus the minimum sucrose destruction ranges from 0% at ME to 0,65% at FX.

An independent non-quantitative indication of inversion is the pH decline across the evaporator, the pH being measured after dilution to the brix of clear juice, and at ambient temperature.

Comparative figures are presented in Table 3 and they show the same general trends as the glucose % sucrose figures (ie the evaporators which produced the most glucose also produced the most acid). Within the pH range involved, if the temperatures and residence times are high enough to cause acid production from invert sugars then these conditions will also cause appreciable sucrose degradation. Furthermore the acidic products will catalyse additional sucrose inversion by lowering juice pH.

Table 2

Increases in glucose % sucrose across stages of different evaporators

Run No.	From CJ to exit 2nd effect						Overall (CJ to syrup)					
	FX	(b) PG−1c	PG+1c	NB	ME	ML	FX	PG−1c	PG+1c	NB	ME	ML
1	0,41	0,03	0,17(c)	0,08	0,08	−0,04	0,45	0,25	0,18	0,04	0,14	−0,02
2	0,35	0,19		0,19	−0,16	0,08	0,28	0,05		0,16	−0,15	0,05
3	0,44	0,07		0,34	−0,02		0,60	0,09		0,51	−0,07	
4	0,25	−0,01		0,18	0,05		0,28	0,07		0,26	0,07	
5	0,17	0,19		0,30	0,06		0,20	0,19		0,37	0,08	
(a) 6	0,40	0,08					0,37	0,06				
7		−0,04						0,07				
Mean	0,34	0,07	0,17	0,22	0,00	0,02	0,36	0,11	0,18	0,27	0,04	0,01

(a) Runs 5 and 6 were done simultaneously at FX but with different throughputs on the two evaporator sets - 5= A set approximately 360 t CJ h⁻¹, 6 = B set with approximately 260 t CJ H⁻¹.

(b) PG−1c represents the PG evaporator running with the 1c vessel by-passed.

(c) Each figure in the table represents the average of 7 to 9 analyses composited from the 14 to 18 shift samples. The PG+1c result is the average of 12 analyses.

Table 3

Decreases in pH across different evaporators
(pHs measured at CJ Bx and ambient temperature)

Run No.	From CJ to exit 2nd effect						Overall (CJ to syrup)					
	FX	PG−1c	PG+1c	NB	ME	ML	FX	PG−1c	PG+1c	NB	ME	ML
1	0,57	(0,51)	0,53	0,02	(0,04)	0,31	0,60	(0,27)	0,50	0,04	(0,19)	0,23
2	(0,77)	0,29		0,81	0,27	0,50	(0,77)	0,24		0,96	0,32	0,48
3	(0,57)	0,32		0,58	0,32		(0,77)	0,30		0,55	0,27	
4	0,56	0,33		0,58	0,25		0,59	0,34		0,57	0,22	
5	0,57	0,50		0,66	0,38		0,54	0,56		0,47	0,30	
6	0,58	0,38					0,57	0,37				
7		0,44						0,51				
Mean	0,60	0,39	0,53	0,53	0,25	0,40	0,64	0,37	0,50	0,52	0,26	0,35

( ) indicates that some atypical CJ pH's have been ignored.
The footnotes to Table 2 also apply to Table 3.

Thermal degradation of invert sugar produces a range of acidic products including acetic acid. Acetic acid in vapour condensates can be easily measured and was therefore monitored as a possible additional indicator of sucrose inversion.

Results are summarised in Table 4 and show striking differences between evaporators, with the general trend agreeing with the other indicators of inversion. FX in particular had high levels of acetic acid with 97% of the V2 samples containing more than 10 mg ℓ^{-1} whereas none of the V2 samples from ME had more than 10 mg ℓ^{-1}. Corrosion of vapour lines has been a problem at FX and it probably reflects the high acidity levels in the vapours.

Theoretical estimates of inversion for PG have agreed well with measured inversion but for FX the theoretical estimates have been considerably lower than measured amounts. The first two effects at FX are Kestners and it is possible that the theoretical estimates were conservative because of erroneous assumptions about conditions in these vessels. The influences of slight changes in assumed conditions are shown in Table 5 and illustrate that the measured inversion of about 0,65% is within the bounds of expectation, especially because calculation of the base case was done without correction for boiling point elevation and with calculated residence times, not measured times. The calculated times are based

Table 4

Acetic acid in vapour condensates from different evaporators
(% of samples with acetic acid exceeding specified concentration)

Factory	Vapour 1						Vapour 2						Vapour 3					
	FX	PG−1c	PG+1c	NB	ME	ML	FX	PG−1c	PG+1c	NB	ME	ML	FX	PG−1c	PG+1c	NB	ME	ML
10 mg ℓ^{-1}	32	25	0	31	3	0	97	22	54	44	0	0	89	0	—	42	0	7
25 mg ℓ^{-1}	0	6	0	11	3	0	79	2	9	6	0	0	39	0	—	3	0	3
No. of Samples	34	48	24	35	33	12	33	46	11	34	33	12	28	48	0	31	33	12

on low juice levels (30 cm in tubes) and they assume negligible residence time in the separators.

Table 5

Some "what if" calculations of inversion in the FX evaporator

Assumed condition	Inversion (%)
1. Base case (a)	0,19 (b)
2. Juice at 230 cm in Kestner tubes	0,35
3. As per '2' but using exit pHs	0,43
4. As per '3' but Kestner temperatures up 2°C	0,52
5. As per '4' but Kestner temperatures up 5°C	0,70
6. As per '5' but retentions calculated from mean in/out volume	0,94

(a) The base case assumptions were (i) inlet pH to each vessel was used in the calculation, (ii) 30 cm juice in tubes, (iii) negligible juice in separators, (iv) residence time in Kestners based on inlet juice volume and (v) juice temperatures derived from vapour pressures without correction for boiling point elevation.

(b) The measured changes in glucose % sucrose indicated inversion of 0,67% when the evaporator was operated under the conditions of flow, pH and brix which were used in these calculations.

General Discussion and Conclusions

The need for efficient utilisation of energy dictates that large amounts of vapour should be bled from the first two effects of evaporators. To achieve this a large portion of the total evaporation has to be done in the first two effects and at temperatures above 100°C. If too much evaporation is done in these effects then there is a danger of combining conditions of low pH and high temperature. The low pH is caused by a combination of factors. Firstly, as evaporation progresses the existing acids become more concentrated and therefore there is a relationship between brix and pH (Figure 2). Secondly, the pH decreases with increasing temperature[4] and thus a combination of high brix and high temperature causes conditions of low pH. If decomposition of invert sugar occurs then the acid end-products further decrease the pH and thereby catalyse additional inversion. Typical pH levels
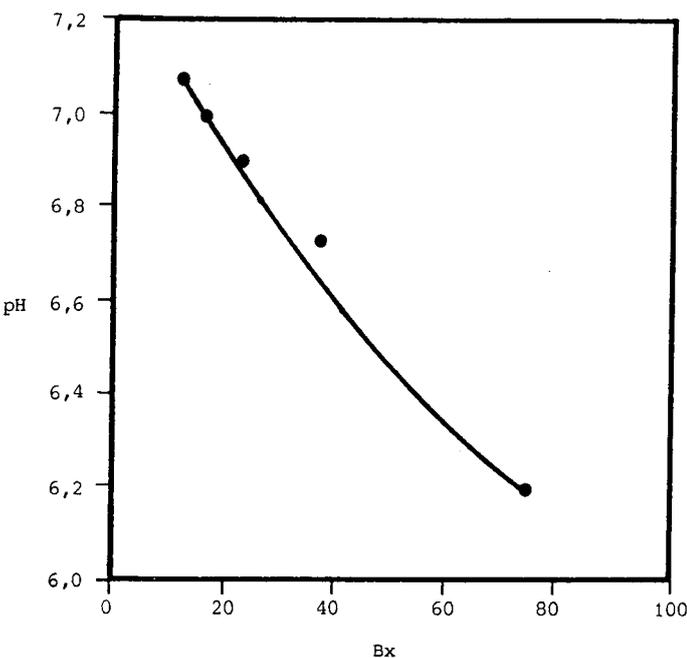
of juice exiting the second effect of the different evaporators are shown in Table 6 and they illustrate that the evaporators in which relatively high inversion has been measured are the ones in which the pH is relatively low in the second effect (and the brix is high).

Table 6

Typical Brixes and pH's of juice exiting the second effect of different evaporators

Factory	FX	PG−1c	PG+1c	NB	ME	ML
Bx	44	28	41	33	24	38
pH at 22°C	6,1	6,5	6,3	6,2	6,8	6,5

Some evaporators are operated under conditions where slight changes can have significant effects. For example, Figure 3 shows that calculated effects that temperature changes would have in the second effect of the PG evaporator (normal pH at 22°C = 6,3, normal temperature = 104°C, normal inversion = 0,16%). An increase of 4°C increases inversion by 50%. If the evaporation could be done in the third effect at 98°C without increasing residence time then inversion would be decreased by 50%. The calculated influence of pH (22°C) in the same PG vessel is shown in Figure 4.

Only two sets of samples were collected from Malelane (ML) and they both showed lower inversion than had been anticipated from the heating surface distribution (Figure 1). When the heating surface in the first two effects was related to cane throughput, instead of the total heating surface, then the results for ML fitted the general trend (Figure 5). With hindsight this method of expressing heating surface is perhaps more logical, particularly when comparing quadruple effect evaporators (ML) with quintuple effects (all others). Staff at ML suspect that the high undetermined loss at ML is caused by packing and warehousing losses, not by evaporator losses. The limited number of evaporator samples from ML tends to confirm this.
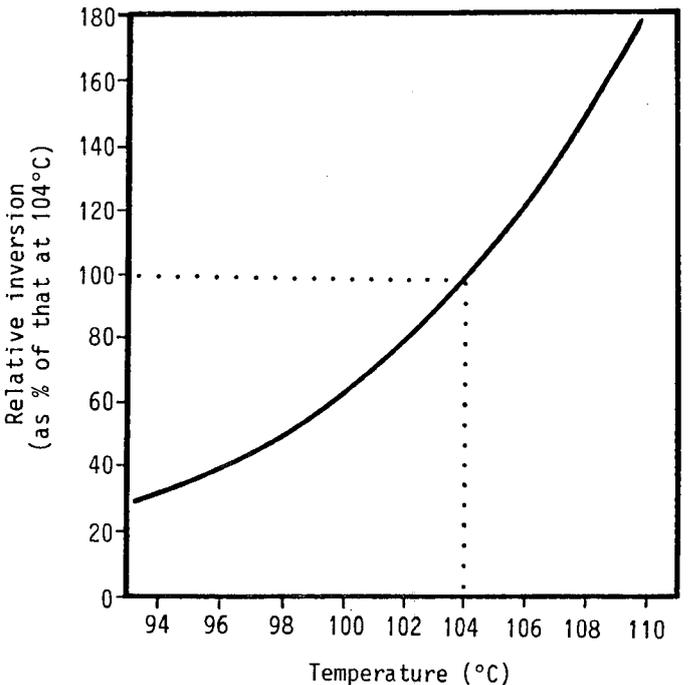

FIGURE 2 The relationship between pH and Bx when a clear juice was evaporated under mild conditions (57°C) in the laboratory.

FIGURE 3 Calculated relative inversion for the second effect of the PG evaporator if operated at different temperatures. (Normal temperature = 104°C; pH = 6,1-6,3 and inversion at 104°C = 0,16%).
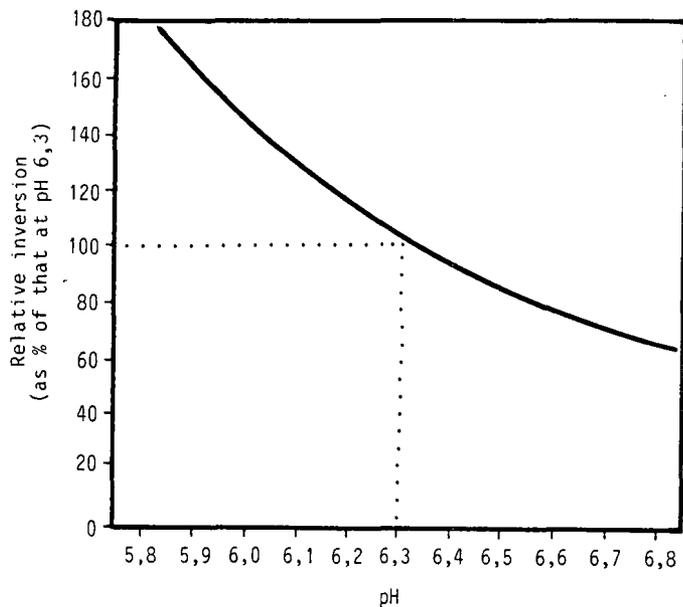
FIGURE 4 Calculated relative inversion for the second effect of the PG evaporator if operated at different pH's. (Normal pH (at 22°C) = 6,3, normal inversion = 0,16%).
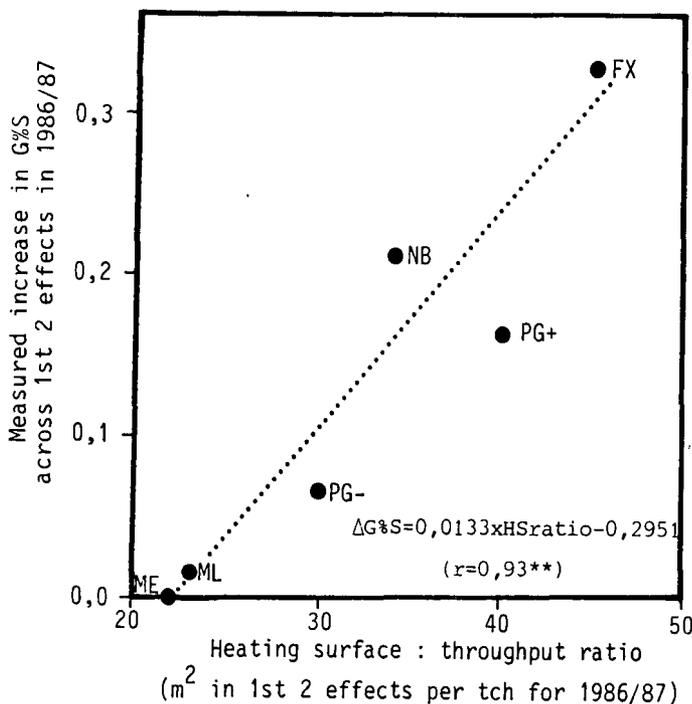

FIGURE 5 Measured increase in glucose % sucrose related to the heating surface in the first two effects of different evaporators.

If the relationship between front end heating surface and sucrose loss (Figures 1 and 5) is a true cause-and-effect relationship then the regression equations could be useful in assessing the optimum level of vapour bleeding so that savings on fuel are not completely offset by losses due to sucrose degradation. However sucrose degradation also causes problems of reduced syrup purity and hence reduced boiling house recovery. The cost of this and of increased colour needs to

be taken into account. Application of the "SJM" formula suggests that inversion equivalent to 0,5% will drop the boiling house recovery by 0,9%.

The fact that juice pH declines as temperature rises has been overlooked in a number of theoretical estimates of sucrose inversion (eg Wittwer[7]) thus causing serious errors and wrong conclusions regarding the economy of extensive vapour bleeding. This study has shown that costly sucrose losses are occurring in some evaporators. At PG, fuel savings due to the use of the 1C vessel and its associated vapour recompressor were estimated to be worth R100 000 per season based on the 1983 coal price of R27 t^{-1} (Allan *et al.*[2]). At present coal prices the saving is worth about R200 000/ season but the equipment probably causes at least 0,5% reduction in boiling house recovery. If sugar is worth R600 t^{-1} then this loss is equivalent to approximately R300 000/ season.

Acknowledgements

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APPENDIX I

Frequency distributions showing the results of some daily pH measurements across the PG evaporator (pH's measured at room temperature and without adjustment of Bx).

pH	C.J.	Out Kest.	Out 1c	Out 2b	Out 3b	Out 4b	Syrup
7.3							
7.2	*						
7.1	********						
7	**********	*					
6.9	*	****					
6.8	*	**					
6.7	*	**	*				
6.6		*	***				
6.5		**	***	***			
6.4		***	**	*	****	****	*
6.3		*****	**		***	**	**
6.2		**	*******	***	****	****	****
6.1			***	**	***	****	*****
6			****	***	***	****	
5.9				*	**	*	**
5.8				*	**	*	*
5.7					*	**	**
5.6					*		*
5.5							

APPENDIX II

Theoretical analysis of inversion in the Pongola Evaporator

Major assumptions

1. Juice brixes and pHs as per samples of 9.11.85.
2. Juice height in tubes = 30 cm.
3. Kestner retention time calculated on inlet volume of juice, 1c based on mean of inlet and outlet, others based on outlet volume.
4. The inlet pH to each vessel is used in the calculations.

Vessel	(CJ)	KEST	1c	2a+2b	3a+3b	4a+4b	5a+5b
Operating volume		12.80	19.00	14.00	7.20	7.20	7.40
Bx at outlet	13.60	21.96	28.32	34.60	41.12	48.80	64.08
Density	1.05	1.09	1.12	1.15	1.18	1.22	1.31
Suc (g mℓ^{-1})	0.14	0.24	0.32	0.40	0.49	0.60	0.84
Tons h^{-1} out	205.00	126.96	98.45	80.58	67.80	57.13	43.51
Tons vap. h^{-1} (calc.)		78.04	28.51	17.87	12.78	10.67	13.62
Vapour as % total		48.33	17.66	11.06	7.91	6.61	8.44
Juice vol h^{-1} out	194.84	116.62	88.06	70.17	57.40	46.75	33.22
Res. time (m)		3.94	11.14	11.97	7.53	9.24	13.37
pH at 22°C	7.00	7.00	6.45	6.30	6.20	6.10	6.10
Delta pH t^{-1} × 100		−1.22	−0.79	−0.69	−0.62	−0.57	−0.57
Operating temp.	102.00	108.00	112.00	105.00	96.00	86.00	62.00
Operating pH		5.95	5.74	5.73	5.74	5.74	5.87

INVERSION

%/vessel		0.04	0.25	0.14	0.03	0.01	0.00

TOTAL % = 0.47