ESTIMATION OF SUCROSE INVERSION IN EVAPORATORS

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Abstract

With the use of high performance ion chromatography, investigations conducted in a number of sugar factories confirmed that glucose/brix or glucose/chloride could be used as a marker to localise and estimate sucrose inversion in evaporators, and establish its contribution to the undetermined loss. In most cases sucrose degradation took place in the later effects, where evaporator tubes were found blocked with hard scales of silica. In others it occurred at the front end of the evaporators where most of the evaporation was done. Sucrose loss ranged from 0,18 to 0,75% sucrose, or 0,015 to 0,081 sucrose % cane, representing 5 to 85% of the undetermined loss.

Keywords: Sucrose inversion, loss in evaporators, undetermined loss.

Introduction

In evaporators, sucrose may be lost either chemically through acid catalysed inversion or physically by vapour entrainment. The main reasons for sucrose inversion are high temperature and long residence time, especially if there is a dead volume in the evaporators, which favours the formation of organic acids from invert sugars. The acids become concentrated across the evaporators and the resulting drop in pH causes sucrose inversion. Mega and Van Etten (1988) demonstrated that acid catalysed hydrolysis of sucrose yields D-glucose and a fructose carboxonium ion which reacts with water to form D-fructose. The fructose carboxonium ion can further react with sucrose to form kestoses. In the first effect of the evaporators, as water is predominant, the formation of fructose is favoured, whereas in the later effects, the high sucrose content probably explains the formation of kestoses.

Sucrose inversion is never monitored directly by the changes in sucrose concentration even if determined by accurate modern techniques such as gas liquid chromatography (GLC), high pressure liquid chromatography (HPLC) or high performance ion chromatography (HPIC), since the actual sucrose loss may be smaller than the analytical error. As a marker to detect and estimate sucrose inversion, Davies *et al.* (1942) used chloride which was claimed not to be destroyed during the process, and Schäffler *et al.* (1985) employed a glucose/brix or glucose/chloride ratio. Purchase *et al.* (1987) used glucose referenced against sucrose and acetic acid formed in the vapour condensates for sucrose loss monitoring, Edye and Clarke (1996) also tried to determine sucrose inversion using glucose/sucrose ratio at lower pH values (pH 25 < 7), glucose is considered more stable than fructose (Sapranov and Koltschewa, 1975), which is more sensitive to heat destruction, as already mentioned.

Experimental

This study was initiated in 1994 at Union St Aubin sugar factory to determine whether sucrose inversion was occurring in the evaporators, and its contribution, if any, to the high undetermined loss experienced. The factory has quintuple effect evaporators consisting of a Kestner in series with a Robert's vessel forming the first effect, and of two vessels in series for the fifth effect. For sampling purposes, the residence time in each evaporator vessel was estimated from the juice flow rates and vessel volumes according to the method of Honig (1963), and were found to approximate to five minutes in all except the last two, where 10 minutes were recorded (Table 1).

Table 1

Estimation of retention time in evaporator effects at Union St Aubin

	E1a	E1b	E2	E3	E4	E5a	E5b
Number of tubes	1 700	4 330	3 095	2 827	2 270	1 952	2 270
Length of tubes (m)	5,48	2,52	2,35	1,93	1,28	1,93	1,28
Diameter of tubes (m)	0,04	0,035	0,035	0,035	0,04	0,05	0,04
Total tube volume (m^3)	11,71	10,50	7,00	5,25	3,65	7,70	3,65
Volume of juice in the tubes* (m^3)	3,90	3,50	2,33	1,75	1,22	2,57	1,22
Volume below the calandria (m^3)	1,62	3,16	0,90	1,37	1,37	1,48	1,64
Total volume of juice in body (m^3)	5,52	6,66	3,23	3,12	2,59	4,05	2,86
Brix in (%)	11,39	15,84	20,20	22,84	27,88	41,70	54,48
Brix out (%)	15,84	20,20	22,84	27,88	41,68	54,50	66,30
Mean brix (%)	13,62	18,02	21,52	25,36	34,78	48,08	60,39
Density of juice at mean brix	1 052	1 071	1 087	1 104	1 149	1 218	1 285
Mass of juice in the body (kg)	5 811	7 134	3 515	3 445	2 973	4 928	3 672
Mass of brix in the body (kg)	791	1 285	756	874	1 034	2 370	2 218
Retention time** (minutes)	3,60	5,90	3,50	4,00	4,70	10,90	10,20

Clarified juice, process liquor from each vessel of the evaporator station and syrup were sampled every 10 minutes with an appropriate time lag (five minutes) between each vessel. Each product was kept in ice-cooled plastic bottles to avoid inversion and evaporation after sampling, which lasted for one hour. Sub-samples of each product were composited, two aliquots were preserved separately with mercuric iodide solution (0,5 mL/litre) and formaldehyde (0,5 mL/100 mL) according to analyses required, and were deep frozen pending analysis.

Chloride was determined on the samples preserved by formaldehyde, using a constant current amperometric-coulometric titrator. Brix (Bx) was measured on mercuric iodide preserved samples, using a conventional refractometer; fructose, glucose and sucrose were determined using a high performance ion chromatograph equipped with a pulsed amperometric detector according to the official ICUMSA method for molasses (Schäffler, 1994), modified for juices and syrup. The pH was determined at room temperature and on undiluted samples.

Three trials, each of one hour duration, were effected on three occasions in 1994 at Union St Aubin sugar factory, and were repeated during the 1995 crop after maintenance had been carried out in the evaporators.

Additional experiments were also conducted at six other factories, mostly in 1994.

Results and discussion

Analytical results

Analytical results are shown in Table 2 for the three trials carried out in the quintuple effects of Union St Aubin sugar factory. The pH profile across the evaporator station (Figure 1) shows a drop from 7,2 to 6,3 mainly due to the increased brix along the evaporators. In contrast, all the other parameters investigated, namely brix, sucrose, fructose, glucose and chloride increased across the evaporators (Figure 2) as the juice became more concentrated.

Table 2

Analytical results of trials carried out at Union St Aubin

	CJ	E1a	E1b	E2	E3	E4	E5a	E5b
Trial 1								
pH	7,200	7,010	6,800	6,670	6,540	6,430	6,35	–
Brix %	12,180	16,020	19,680	24,020	29,200	39,000	54,520	66,950
Sucrose %	10,940	13,310	15,610	17,980	26,150	35,050	44,100	55,740
Fructose %	0,122	0,170	0,205	0,245	0,401	0,486	0,616	0,796
Glucose %	0,122	0,158	0,193	0,232	0,386	0,481	0,619	0,798
Chloride %	0,086	0,107	0,135	0,160	0,207	0,284	0,386	0,468
Trial 2								
pH	7,550	7,520	7,280	7,030	6,920	6,860	6,730	6,550
Brix %	11,390	15,840	20,200	22,840	27,880	41,680	54,480	66,300
Sucrose %	10,300	12,830	15,390	18,040	25,030	37,310	45,690	56,060
Fructose %	0,122	0,160	0,204	0,235	0,264	0,513	0,619	0,822
Glucose %	0,120	0,155	0,202	0,230	0,357	0,503	0,637	0,821
Chloride %	0,065	0,092	0,116	0,127	0,164	0,231	0,338	0,370
Trial 3								
pH	7,640	7,480	7,250	7,140	6,990	6,900	6,840	6,720
Brix %	11,390	15,130	18,920	21,300	26,800	38,080	50,520	66,200
Sucrose %	9,780	12,890	15,460	16,950	24,030	33,600	44,120	56,050
Fructose %	0,109	0,130	0,179	0,198	0,288	0,401	0,554	0,688
Glucose %	0,110	0,163	0,178	0,196	0,296	0,430	0,556	0,715
Chloride %	0,066	0,084	0,113	0,122	0,156	0,209	0,291	0,375

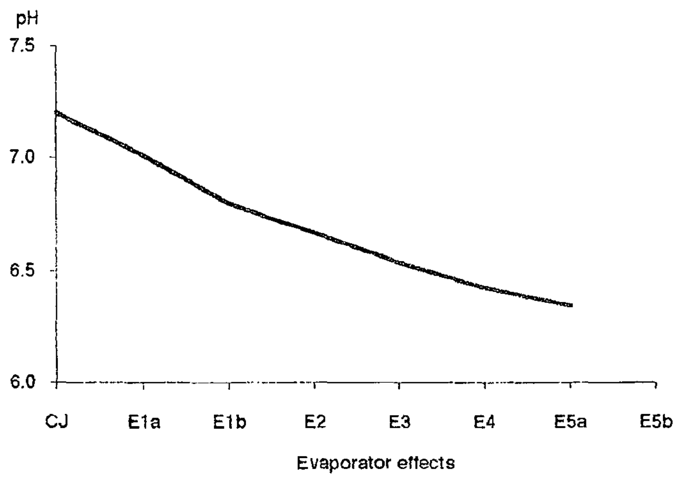

FIGURE 1: pH drop across the quintuple effect of Union St Aubin (trial 1)

Marker for sucrose inversion

The mean fructose/glucose (F/G) ratio for the three trials across the evaporator effects is compiled in Table 3. Generally, this ratio decreased across the effects, indicating the destruction of fructose relative to glucose which is more stable to heat decomposition. Glucose decomposition is small; which is the reason why sucrose (S) loss estimation using glucose as a marker may be underestimated. However, up to now, there is no better marker than glucose for the determination of sucrose inversion.

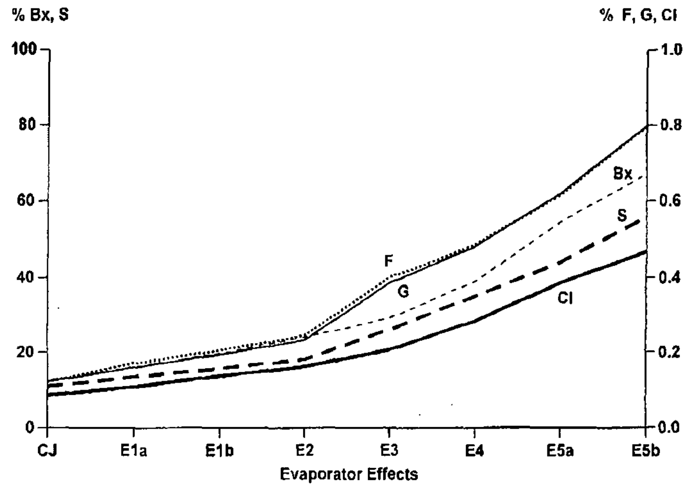

FIGURE 2: Changes in % brix, sucrose, fructose, glucose and chloride across the quintuple effect at Union St Aubin (trial 1)

Table 3

Average F/G ratios across the evaporator effects at Union St Aubin

	CJ	E1a	E1b	E2	E3	E4	E5a	E5b
F/G	1,00	1,03	1,02	1,03	1,01	0,98	0,98	0,98

Detection of sucrose inversion

Sucrose inversion was estimated by reducing sugars/pol ratio before the advent of modern instrumental techniques such as GLC or HPIC, which enables the separate determination of glucose and fructose. The average data from the three trials at Union St Aubin compiled in Table 4 clearly illustrate that there is an increase in (F+G)/S ratio in the third effect, confirmed by all other ratios of G/S, F/Bx, F/Cl, G/Bx, and G/Cl.

Table 4

Average (F+G)/S, G/S, F/Bx, F/Cl, G/Bx and G/Cl ratios across the evaporator effects at Union St Aubin

	CJ	E1a	E1b	E2	E3	E4	E5a	E5b
(F+G)/S	1,00	1,05	1,10	1,10	1,22	1,17	1,19	1,21
G/S	1,00	1,07	1,09	1,10	1,21	1,17	1,20	1,22
F/Bx	1,00	0,97	0,99	0,97	1,23	1,16	1,12	1,14
F/Cl	1,00	0,95	1,00	1,03	1,18	1,11	1,03	1,11
G/Bx	1,00	1,01	0,97	0,96	1,22	1,18	1,13	1,16
G/Cl	1,00	1,04	0,96	0,99	1,21	1,20	1,10	1,18

Changes in G/S, G/Bx and G/Cl ratios for trials 1, 2 and 3 are shown in Figures 3, 4 and 5 respectively. G/Bx and G/Cl ratios appear to be better markers than G/S, which fluctuates considerably. Figures 3 and 4 indicate inversion occurring in E3, and Figure 5, in E3 as well as in E1a.

The ratios of Bx/Cl in clarified juice and syrup determined at various factories (Table 5), are not significantly different at P=0,05. The constant Bx/Cl relationship implies that brix is not preferentially destroyed to chloride, and that either brix or chloride can be used as reference.

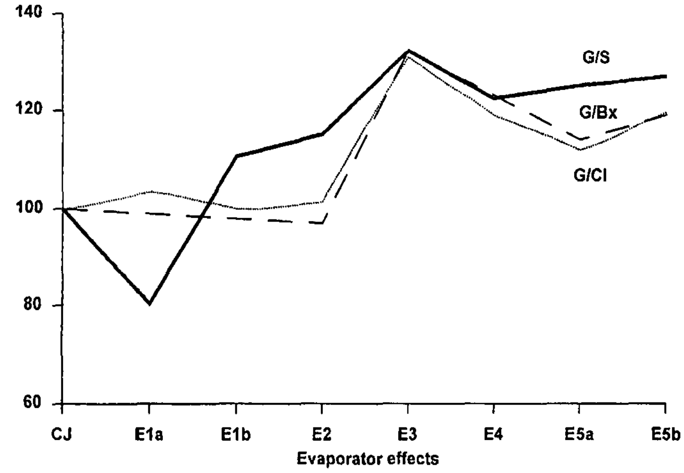

FIGURE 3: Changes in G/Bx, G/Cl and G/S ratios across the evaporators at Union St Aubin (trial 1) normalised with their respective ratios of clarified juice as reference (100)

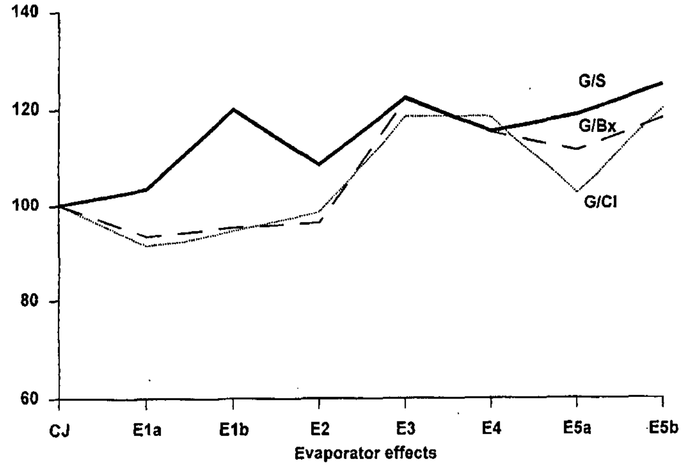

FIGURE 4: Changes in G/Bx, G/Cl and G/S ratios across the evaporators at Union St Aubin (trial 2) normalised with their respective ratios of clarified juice as reference (100)

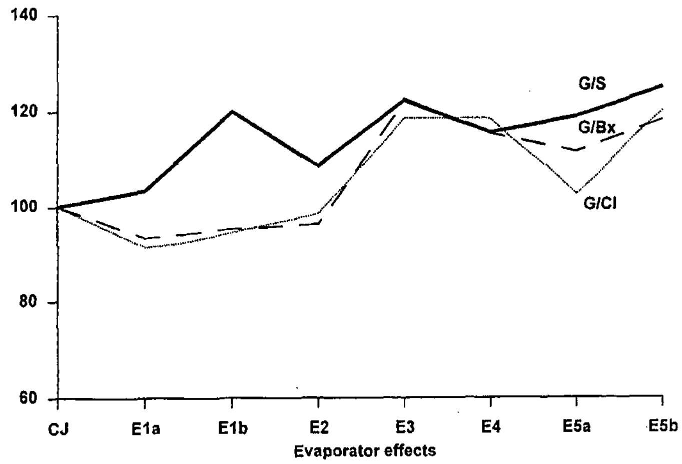

FIGURE 5: Changes in G/Bx, G/Cl and G/S ratios across the evaporators at Union St Aubin (trial 3) normalised with their respective ratios of clarified juice as reference (100)

Table 5

Bx/Cl ratios of clarified juice and syrup determined at various factories

Factory	Bx/Cl ratios	
1994	Clarified juice	Syrup
Britannia (trial 1)	207,2	196,9
Britannia (trial 2)	216,2	210,4
Union St Aubin (trial 1)	142,5	143,1
Union St Aubin (trial 2)	174,4	178,2
Union St Aubin (trial 3)	173,9	176,5
DRBC (trial 1)	196,5	190,6
DRBC (trial 2)	194,1	181,9
SSRR	135,0	131,9
1995		
St Félix	131,9	117,0
Britannia	170,3	173,9
Union St Aubin	147,3	149,5
DRBC	148,1	142,2
MDA	151,7	154,7

SSRR = *Sociétée de Sucrière de Rivière du Rempart*
DRBD = *Deep River Beau Champ*
MDA = *Mon Désert Alma*

Quantitative estimation of sucrose inversion

There have been few studies on sucrose inversion in evaporators. Vukov (1965) described the estimation of sucrose inversion in an individual evaporator effect using the residence time and the reaction rate constant, given the pH at the operating temperature.

Schäffler *et al.* (1985) measured sucrose inversion indirectly by the increase in glucose/brix or glucose/chloride ratio:

% sucrose inversion =

$$\frac{\left[\frac{\%G}{Bx}\right] \text{out} - \left[\frac{\%G}{Bx}\right] \text{in}}{\left[\frac{\%S}{Bx}\right] \text{in} \times MW_G} \times MW_S \times 100$$

or

% sucrose inversion =

$$\frac{\left[\frac{\%G}{Cl}\right] \text{out} - \left[\frac{\%G}{Cl}\right] \text{in}}{\left[\frac{\%S}{Cl}\right] \text{in} \times MW_G} \times MW_S \times 100$$

where MW_S = molecular weight of sucrose – 342
MW_G = molecular weight of glucose – 180.

Purchase *et al.* (1987) employed a glucose/sucrose ratio in place of a glucose/brix or glucose/chloride ratio:

% sucrose inversion =

$$\frac{\left[\frac{\%G}{S}\right] \text{out} - \left[\frac{\%G}{S}\right] \text{in}}{MW_G} \times MW_S \times 100$$

$$= 190 \left[\left[\frac{\%G}{S}\right] \text{out} - \left[\frac{\%G}{S}\right] \text{in}\right]$$

In order to calculate the sucrose loss in a particular evaporator effect, the data at the inlet and outlet of that effect are considered, and to calculate the cumulative loss from clarified juice to a particular evaporator vessel, the data from the effect are normalised with those of the clarified juice as reference.

Location and magnitude of sucrose loss

Typical results of % sucrose loss in clarified juice across the evaporators (cumulative) and % sucrose loss in a particular effect (individual) are depicted in Figure 6 for trial 1 carried out at Union St Aubin, where G/Cl was used as the marker. It can be seen that sucrose loss occurred in the third effect and amounted to 0,71% sucrose. Similar experiments carried out at Deep River Beau Champ (DRBC) sugar estate and Médine showed that sucrose inversion took place in the second and fourth effects and amounted to 0,42 and 0,75% sucrose respectively (Figures 7 and 8).

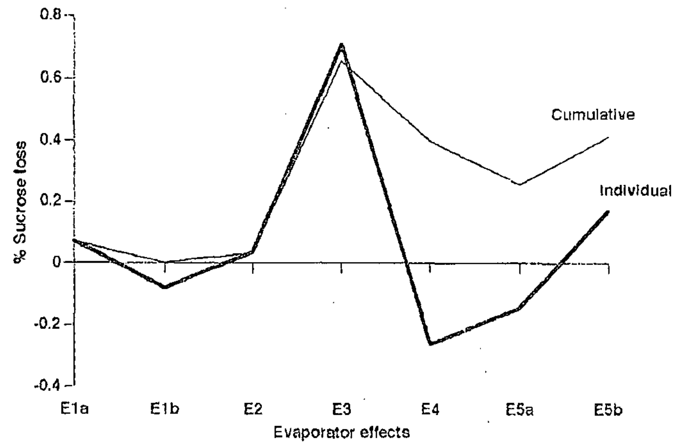

FIGURE 6: Sucrose inversion across the evaporators at Union St Aubin – trial 1 (G/Cl as marker)

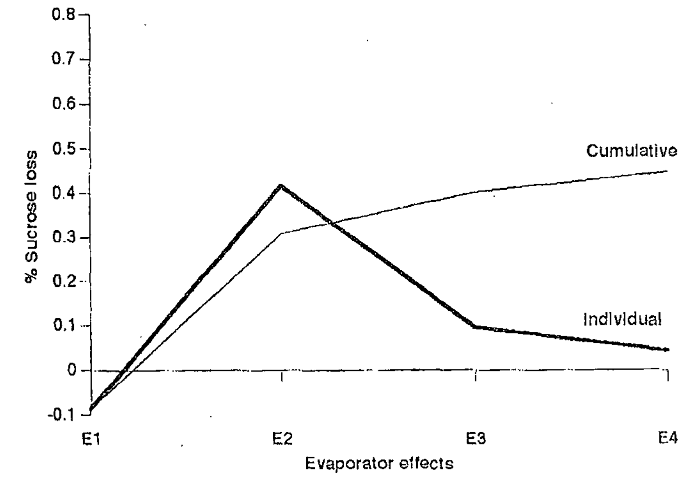

FIGURE 7: Sucrose inversion across the evaporators at DRBC – (G/Cl as marker)

Table 6 summarises the location and magnitude of sucrose loss in evaporators at various factories. All sucrose inversions were calculated from G/Cl ratios except in the case of Médine where G/Bx ratios were used. Table 6 also indicates the location where vapour bleeding was effected and the percentage of the heating surface of the bled vessels to that of the evaporators. From Table 6 it can be seen that sucrose inversion occurred most often in the third/fourth effects, and also in the first/second effects from which vapour bleeding was in operation. The heating surfaces in these bled vessels constituted 46-100% of the total evaporator heating surface, and may explain why inversion was taking place there. The sucrose loss in the effect concerned ranged from 0,18 to 0,75% at the factories investigated.

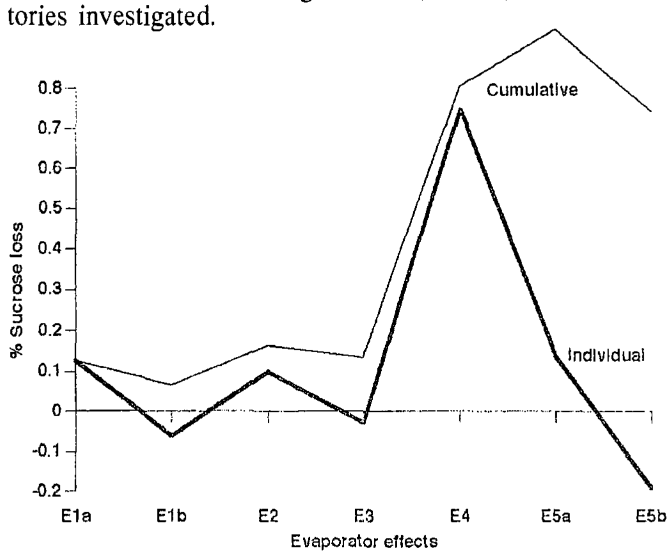

FIGURE 8: Sucrose inversion across the evaporators at Médine – (G/Cl as marker)

Purchase *et al.* (1987) found that the sucrose degradation in the evaporators at Pongola was 0,5%, with almost all the loss occurring in the first two effects of the evaporator, where a large portion of the evaporation was done, with vapour being recompressed for re-use in one of the vessels. They found that the energy savings realised by the extensive vapour recompression were worth less than the consequential destruction of sucrose. They also found a correlation between the relative size of the first two evaporator effects and the undetermined loss.

Table 6

Location and magnitude of sucrose loss (G/Cl or G/Bx reference) in evaporators, locations of vapour bleeding, % heating surfaces of the bled vessels to the total evaporator heating surfaces at various factories

Factory	Year	Sucrose loss		Location of vapour bleeding	Relative heating surface of bled vessels to that of the evaporators %
		Location	% sucrose		
Union St Aubin	1994-1 1994-2 1994-3 E3	E3 E3 E1a 0,39	0,71 0,49 0,34	E1, E2	57
DRBC	1994	E2	0,42	All effects E1-E3	100
Médine	1994	E4a	0,75	All effects E1-E4	100
SSRR	1994 E4	E3 0,59	0,48	E1, E2, E3	66
St Félix	1995 E3	E1 0,18	0,19	E1	46

SSRR = Société de Sucrière de Rivière du Rempart
DRBC = Deep River Beau Champ

At Union St Aubin, it was not clear why sucrose inversion took place in the third effect evaporator. From the experience gathered at other factories, especially at Médine, it would not be surprising that the same explanation (detailed below) is applicable.

At DRBC, vapour bleeding was done in all effects, and sucrose degradation occurred in E2, which is probably the expected consequence of extensive bleeding.

At Médine, sucrose degradation took place in the E4a vessel. During weekend shut-down, it was observed that there were appreciably more scales in the two fourth effects than in other vessels, and some of the evaporator tubes in the fourth vessel were blocked with hard silica-based scale. The application of antiscalant is already practised at Médine to alter the structure of the scales so that they become softer and more easily cleaned, and increased dosage of the antiscalant used is envisaged.

The evaporator situation at Sociétée Sucrière de Rivière du Rempart (SSRR) was similar to that at Médine. Sucrose destruction occurred in E3 and E4, where heavy scalings were again observed.

Table 7

Sucrose loss across the evaporators expressed as % sucrose in clarified juice, (G/Cl or G/Bx reference) as % cane and its relationship with undertermined loss

Factory	Year	Sucrose loss evaporator		Undetermined loss Sucrose % cane	Contribution of inversion to undetermined loss %
		% sucrose in clarified juice	% cane		
Union St Aubin	1994-1	0,41	0,045	0,25	18
Union St Aubin	1994-2	0,44	0,048	0,27	18
Union St Aubin	1994-3	0,29	0,032	0,27	12
Union St Aubin	1995	0,14	0,015	0,30	5
DRBC	1994	0,45	0,050	0,37	14
Médine	1994	0,75	0,081	0,29	28
SSRR	1994	0,66	0,073	0,00	-
Britannia	1994	0,30	0,033	0,04	83
Britannia	1995	0,27	0,030	0,04	75
MDA	1995	0,41	0,045	0,08	56
St Félix	1995	0,30	0,033	0,22	15

SSRR = Société de Sucrière de Rivière du Rempart
DRBC = Deep River Beau Champ
MDA = Mon Désert Alma

At St Félix, vapour bleeding was effected from E1, which was oversized. The heating surface was 1 164 m^2 for a factory of 60 t cane/hour. At one weekend cleaning, the mass of scales found in E1, E2, E3 and E4 were 24, 3,5, 20 and 4 kg respectively. The fact that there were more scales in E3 confirmed long residence time, sucrose inversion, and more scale deposition, which entailed further inversion.

The sucrose inversion in evaporators in terms of % sucrose in clarified juice can be converted to % cane by multiplying by 0,11, if the assumption is made that sucrose content in cane is 11%. The undetermined losses at factories concerned and the contribution of the inversion loss to the undetermined loss are given in Table 7. At Union St Aubin, inversion was only about 18% of the undetermined loss in 1994, which was further reduced to 5% after maintenance was performed. At DRBC, Médine and St Félix, the inversion loss constituted 14, 28 and 15% respectively, of the undetermined loss. This percentage was high at Britannia and Mon Désert Alma (MDA), where the undetermined losses were quite low.

Conclusions

Glucose/brix or glucose/chloride ratios have been successfully used to localise and measure sucrose degradation in evaporators at a number of sugar factories. Most losses occurred in the third/fourth vessels where there was appreciable scaling and blocking of evaporator tubes resulting in longer residence time, sucrose inversion and more scale deposition, which entailed further inversion; in other cases, losses took place in the first two vessels where there was extensive vapour bleeding. The contribution of sucrose degradation to undetermined loss could be assessed.

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