

PILOT PLANT INVESTIGATION OF CLEAR JUICE SOFTENING

SB DAVIS, SD PEACOCK AND DC WALTHER

Sugar Milling Research Institute, University of Natal, Durban, 4041

Abstract

The benefits of clear juice softening in the raw house, such as reduced evaporator scaling, have been discussed previously. The effects on boiling house operation, including exhaustion, need to be quantified for a full economic evaluation to be performed. Mill clear juice was pumped through a pilot softening unit and evaporated to syrup in the Felixton (FX) evaporator pilot plant. Extended evaporator fouling tests on raw and softened clear juice showed greatly reduced fouling from the softened juice. Batches of raw and softened syrup were boiled in the Sugar Milling Research Institute (SMRI) pilot pan to produce A- and B-massecurites and molasses. The effects of softening on operational parameters such as purity are discussed.

Keywords: juice softening, evaporator, scale, fouling

Introduction

Why use juice softening?

A disadvantage of using lime for mixed juice clarification is the significant residual concentration of calcium and magnesium ions in the juice. Calcium forms the major part of scale in the evaporators, and leads to reduced factory efficiency and increased costs. Juice softening by ion exchange is a relatively simple method of removing calcium and magnesium, and the benefits are well known (Thompson, 1994a; Kwok, 1996). These are:

- Reduced factory downtime for cleaning.
- Reduced cleaning costs, whether by chemical or mechanical means.
- Higher average evaporator throughput as a result of higher average heat transfer coefficients.

However, on the negative side, regular regeneration of the resin is needed and, if brine is used for this purpose, a significant cost is incurred. This includes the direct cost of the salt and the indirect cost of disposal or treatment of the effluent. The disadvantage can be eliminated by using soft molasses for regeneration, as discussed by Kwok (1996).

How does softening work?

The mechanism by which softening works has been well explained by Thompson (1994a), and can be summarised as follows. Cations are held by a cation exchange resin with different strengths according to charge and solvated ion size.

Thus Ca^{2+} is held more strongly than K^+ (higher charge for a similar ion size), and K^+ is held more strongly than Na^+ (smaller solvated ion size for the same charge). However, ion exchange is a dynamic process, and the equilibrium thereof is affected by the concentrations of the different species in solution and on the resin. This enables regeneration to occur as the calcium and magnesium ions on the resin are displaced by the very large number of sodium ions in the regeneration brine.

Conventionally, sodium chloride has been used for regeneration as it is cheap and readily available, but disposal of the effluent presents a problem. The high levels of potassium in molasses have led to other methods of regeneration being proposed. Soft molasses, with low calcium and magnesium concentrations but high sodium and potassium concentrations, can be used for regeneration. These methods were reviewed by Thompson (1994b).

Previous work by Thompson (1994a) at the Sezela (SZ) mill proved that evaporator scale could be reduced and heat transfer coefficients improved by the softening of juice. Pilot pan boiling tests showed a lower exhaustion with softened syrup because of the higher sodium content. However, the effects on a factory boiling house are more complex because of the recycling of massecurites and low grade sugars, and further work was necessary to determine the effects. A further study was therefore undertaken to investigate the operation on a pilot scale of a softening plant, and to examine more closely the effects that softening would have throughout the boiling house.

Experimental

A major benefit of softening is a significant reduction in evaporator fouling. To measure this effect on a pilot scale, it was necessary to run the FX pilot plant evaporator (Walthew *et al.*, 1995) on a feed of softened juice for at least 100 hours. As the products of this evaporation would be used for further testing, it was considered convenient to achieve an output syrup concentration of at least 60° brix from the pilot evaporator. A low feed flow rate of two litres per minute was chosen to achieve a 60° brix in one pass. This rate was well below the resin manufacturer's maximum recommended flow rate for this application in terms of bed volumes per hour of feed. To minimise process disruptions during the softening test, the resin columns were sized to allow for more than 100 hours of continuous operation before exhaustion, thus requiring no regeneration during the test.

For ease of transport and assembly, the pilot softening plant consisted of four mild steel columns of 190 mm in diameter and 1,5 m in height, connected in series. The columns were packed with a total volume of 175 litres of Purolite C-150 macroporous strong acid cation exchange resin in sodium form. Juice withdrawn from the clear juice tank was pumped through a coarse strainer and fed to the inlet of the first softening column. The softened clear juice exiting the last column was fed to the pilot plant evaporator to produce softened syrup. A sample of 200 litres of this syrup was collected for boiling tests in the pilot pan at the SMRI. An untreated control sample of factory syrup was collected from the outlet of the FX syrup pumps.

Two batches of raw (untreated) syrup and two batches of softened syrup were boiled in the SMRI pilot pan, with slurry seeding. The centrifugal run-offs from the two raw syrup boilings were combined and the run-offs from the two softened syrup boilings were combined. The sugars produced were affinated in refined sugar syrup and air dried before analysis. The composite A-run-offs were boiled again to produce hard and soft B-massecurites, and the resulting B-sugars were affinated before analysis. The B-massecurites were difficult to cure, and many fine sucrose crystals passed through the centrifugal screen. Further sucrose crystallised on these crystals in the B-run-offs during storage. For these reasons, the results for the B-boilings were not considered meaningful, and are omitted from this paper. Furthermore, slurry seeding, as practised with the operation of the SMRI pilot pan, is not suitable for B-boilings, and a more appropriate method must be used.

Results

Column fouling and general performance

No problems were encountered with the operation of the softening columns once they were commissioned. The pressure drop across the four columns was initially 600 to 700 kPa when cold, but this dropped to 200 to 300 kPa under normal conditions with hot unsoftened juice. The pressure drop was maintained for 90 hours of operation, and then rose rapidly overnight to 600 kPa. Upon investigation, a layer of mud was found on the top of the leading column, and it is believed that a surge in a clarifier led to a mud overflow that caused the rapid increase in pressure drop. Apart from this, no fouling of the columns was encountered, despite having no prefiltration other than the existing coarse strainer fitted to the evaporator pilot plant.

Softening performance

Catch samples were taken of the incoming hard clear juice and the soft syrup from the pilot evaporator on each of the two days during which syrup was collected for pan boiling. The samples were analysed for cations, ash and colour, with the results shown in Table 1.

The second sample of hard clear juice had a particularly high sodium content. The reason for this is not known, but contamination of the sample by column product was not

Table 1
Cation and ash concentrations in clear juice and soft syrup.

Sample	Concentration (ppm on brix)				Ash (% on brix)
	Ca	Mg	Na	K	
Hard juice 1	2 457	1 365	410	10 237	5,1
Hard juice 2	1 939	916	3 787	7 990	4,6
Soft syrup 1	31	10	11 082	11	4,0
Soft syrup 2	42	17	17 170	11	4,2

possible, as the sample was taken from the factory clear juice line. As expected, calcium and magnesium were almost completely removed from the juice. The residual quantities arose from complexed calcium and magnesium that are not susceptible to adsorption on the resin. As previously discussed, cations are held on the resin with differing strengths, in the order $\text{Ca}^{2+} > \text{Mg}^{2+} > \text{K}^{+} > \text{Na}^{+}$, with sodium being the least strongly held. With the resin initially being in the sodium form, calcium, magnesium and potassium were removed from the juice with displacement of sodium into solution. Should all the sodium have been displaced from the resin, calcium and magnesium would then have displaced potassium from the resin. It can be seen from the juice and syrup analyses in Table 1 that potassium was strongly removed by the resin, and that no elution of potassium seems to have occurred. This indicates that the resin columns were far from being saturated with calcium and magnesium when the samples were taken.

Ash levels decreased slightly, as a result of the relative equivalent mass of sodium compared with the other cations. This would not be so for a column regenerated with soft molasses, as calcium and magnesium from the juice would displace potassium, which has a higher equivalent mass, into solution.

Evaporator fouling

To examine the effect of softened juice on scale formation and composition, samples were collected after each test for analysis by x-ray fluorescence (XRF) and x-ray diffraction (XRD) (Walthew and Turner, 1995). The main components are given in Table 2. These components can be converted into compounds using a simple mass balance procedure (Walthew, 1996a) and the result is given in Table 3. Also shown in these tables are the compositions of scale obtained from a similar experiment performed by Thompson (1994a) using a pilot plant at SZ.

The results in the tables show that:

- raw FX juice contains significantly more silica than raw SZ juice and consequently more silica is found in FX scale
- when softened, the scales produced in the FX and the SZ pilot plants are very similar, with silica being the major component
- significantly smaller amounts of phosphate are precipitated from the 'soft' juice

- amorphous organic material and amorphous phosphate as a percentage of dry scale are significantly reduced when softened juice is used. (The amount of calcium phosphate in the scale from the evaporation of soft juice was higher than expected.)

Table 2

Composition in terms of the main components as a percentage of dry scale (LOI = loss on ignition).

Trial		SiO ₂	CaO	MgO	P ₂ O ₅	Oxalate	LOI
FX pilot plant	soft juice	68	5	1	5	1	21
	hard juice	33	16	5	12	1	31
SZ pilot plant	soft juice	71	1	0	1	1	18
	hard juice	1	22	5	24	1	47

In both experiments the juice brix was raised to between 50 and 60° and therefore represents the change in brix over an entire evaporator train, yielding effective 'composite' scale samples. In practice the difference in scale composition would be most marked in the early effects where calcium phosphate is normally the predominant phase, but would be less noticeable in later effects where silica is more common.

Implications for cleaning

The amount of soft, easy to clean scale (amorphous organic compounds plus amorphous phosphate) was dramatically reduced when softened juice was evaporated. From the point of view of cleaning this could present a problem, since the presence of soft scale is necessary for effective cleaning methods. Although considered a hard scale, silica can be deposited in many forms, including soft chalky type deposits and hard glass-like scale, and only long term trials will determine the type of silica scale formed from softened juice. The scales from both the SZ experiment and the FX trial were encouragingly soft and chalky, and not particularly difficult to remove. The experience of Kwok (1996) also suggests that the silica scale formed may not be particularly problematic. The deposition of silica in evaporators

is a complex process dependent on operating conditions, chemical makeup and factors such as pH control. Dispersants such as polyphosphates may be effective in preventing excessive or hard silica deposits (Walthew, 1994). It would seem, however, that caustic cleaning will remain the most effective method of cleaning if softened juice is used.

Amount of scale formed

The degree of fouling can be quantified in terms of fouling resistance by the equation below, which relates fouling resistance to heat transfer coefficient (HTC) measured at a particular time.

$$R_f = 1/U(t) - 1/U(0),$$

where $U(t)$ is the HTC after the evaporator has operated for a time t , $U(0)$ is the initial HTC when the evaporator is clean, and R_f is the fouling resistance. To make meaningful comparisons between different fouling resistances, the HTCs must be measured under similar operating conditions. Those used in this study are shown in Table 4. R_f is related to thermal conductivity and scale thickness by:

$$R_f = x/k,$$

where x = scale thickness and k = thermal conductivity of scale. The difference in scale thickness can be calculated from measured fouling resistances if the thermal conductivity is assumed to be the same in both cases.

The fouling resistances were calculated to be 0,10 m² °C/kW for softened juice and 0,21 m² °C/kW for raw juice (see Appendix). Assuming the same thermal conductivity (k), the relationship between the scale thicknesses (x) for the soft and raw juices is:

$$x_{(\text{soft})} = 0,5 x_{(\text{raw})}.$$

This suggests that the quantity of scale produced by soft juice will be about 50% of that 'normally' produced. This result agrees with the measured masses of scale removed from the tubes after cleaning, being 7 g after the trial with soft juice, and 18 g after the trial with raw juice.

These results are consistent with the experience of Kwok (1996) who found that scale was reduced by about 50% when using softened juice. However, the quantity formed is higher than that

Table 3
Estimated compounds as a percentage of dry scale.

Trial		Amorphous calcium phosphate	Magnesia hydrate	Silica hydrate	Oxalate	Organic material	Loss on ignition	
							Estimated	Actual
FX pilot plant	soft juice	13	1	88	1	0	24	21
	hard juice	32	7	43	1	15	35	31
SZ pilot plant	soft juice	3	0	93	2	3	26	18
	hard juice	64	7	2	1	27	42	47

Table 4
Conditions of operation.

Feedrate	1,6 kg/min
Delta T	6,7°C
Feed juice temperature	102°C
Feed brix	13-17° brix
Product brix	45-60° brix
Juice height (% total tube length), measured by pressure difference	6-10%
Recycle	Open

expected from a projection using current scale analysis, where it would be expected that the quantity of scale formed would be about one third of that 'normally' experienced (Walthew, 1996b).

Possible sources of error

The brix of the outlet syrup during the raw trial could not be maintained at 55 to 60° brix, as in the softened juice trial, without reducing the flowrate. Changing the flowrate would have made comparison of HTC values unrealistic. Thus for the raw juice trial the syrup brix was typically 45 to 55° brix. This artificially improved the evaporator performance as the lower syrup viscosity resulted in improved heat transfer coefficients as compared with the higher viscosity soft syrup.

The scale from the soft juice evaporation contained a higher concentration of calcium than would be expected from the calcium levels in the juice feed. This was thought to have arisen from contamination with old scale from previous experiments that was still present in the evaporator system. Thorough cleaning of the system will be done before future tests to prevent any contamination of scale samples.

Pilot pan boilings

Selected analytical results of the products of the pan boilings are shown in Table 5. There was no significant difference in behaviour between the boilings performed on the hard syrup and the soft syrup, with comparable brix and pol levels being achieved in the pan boilings. As expected, calcium and magnesium concentrations in the soft products were much lower than those in the hard products. The ion exchange resin used for softening was initially in the sodium form, and hence the sodium levels in the soft products are considerably higher than in the hard products. It must be remembered, though, that the proposed softening scheme would use soft molasses for regeneration of the resin, which would therefore be largely in the potassium form. Hence the softened juice and syrup would have low levels of calcium and magnesium, higher than normal levels of potassium, and the sodium levels would not be greatly changed.

Cation transfer to the crystals during pan boilings was found to vary greatly between hard and soft products, but this is not unexpected given the cation concentration differences of orders of magnitude between the hard and soft products. Despite these large differences, the sulphated ash levels in the hard and soft materials were essentially the same. Exhaustion during boiling is typically characterised by the molasses target purity, which is calculated using the reducing sugars to ash ratio. However, softening does change the relative proportions of the cations, increasing the concentrations of the monovalent cations, which are regarded as being more melassigenic than the divalent cations. Thus, although the target purity formula would indicate that similar exhaustions could be achieved, conventional wisdom predicts that lower exhaustions would be achieved for softened molasses. Difficulties in boiling B-sugars in the pilot

Table 5
Results of pan boilings of hard and soft syrups (where applicable, analyses are reported on brix).

Analysis	Feed syrup		A-sugar		A-molasses	
	hard	soft	hard	soft	hard	soft
Brix	63,1	53,5	–	–	83,8	84,7
Pol	51,6	44,0	–	–	57,9	57,4
Ca (ppm)	2 063	31	12,8	2,0	3 424	84
Mg (ppm)	1 498	17	3,3	0,1	3 053	22
Na (ppm)	246	9 739	0,0	18,1	506	19 254
K (ppm)	7 303	7	14,3	0,0	18 268	28
Sulphated ash (%)	4,2	4,2	–	–	7,9	7,4
Colour	25 920	32 780	325	381	–	–
True purity (%)	82,6	83,0	–	–	68,2	66,7

pan and the inability to boil C-sugars in this study meant that no conclusion could be reached as to the effect of softening on molasses exhaustion. In addition, the sodium to potassium ratio was not what would normally be expected in a full scale installation, and further work in this regard needs to be done.

Conclusions

The softening columns operated without problems for an entire week, and very little fouling of the columns with particulate matter was encountered, despite having no prefiltration other than an existing coarse strainer. Care must be taken that carry-over of mud from the clarifiers does not cause problems. Almost total removal of calcium and magnesium in the feed juice was achieved. The results of scale analysis from the pilot plant evaporator showed a significant decrease in calcium, amorphous phosphate and organic material levels in the scale from the softened juice, with silica becoming the major component. This could make cleaning more difficult, as less 'soft' scale is present. The quantity of scale produced from the softened juice was calculated to be about 50% of that formed from the raw juice. This agrees with previous work on softening.

There was no significant difference in the behaviour of the raw and soft syrups during pan boiling. While the sulphated ash levels were the same in the hard and soft products, the calcium and magnesium concentrations in the soft products were much lower than those in the hard products. This could result in lower exhaustions being achieved in softened molasses. However, difficulties in boiling B- and C-sugars in this study meant that no conclusions could be reached on the effect of softening on final molasses exhaustions.

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APPENDIX 1

Calculation of fouling resistances

Initial HTC in both cases: 2,2 kW/m² °C.

HTC after 100 hours of operation:

Softened juice: 1,8 kW/m² °C.

HTC after 100 hours of operation:

Raw juice: 1,5 kW/m² °C.

Fouling resistance = x/k

= $1/1,8 - 1/2,2$ = 0,10 m² °C /kW softened juice

= $1/1,5 - 1/2,2$ = 0,21 m² °C /kW raw juice.