Possibilities of Ion Exchange Softening of Cane Clarified Juice

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Paper presented at
Sugar Processing Research Institute, Inc.
Atlanta, Georgia
April 4- 7, 2004

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Abstract

Softening of sugar juices and molasses has become an established unit operation in the beet sugar industry. Being a necessary prerequisite for molasses chromatographic separation technology, thin juice ion exchange softening positively affects downstream operations. It eliminates the need for descaling evaporators and improves efficiency of heat transfer. The softening process also improves boiling characteristics of juice and has a positive effect on sugar quality. Cane juice typically has higher hardness than beet thin juice, which means that larger softening systems could be required and more regenerant will be produced. Also the presence of higher suspended solids in cane juice makes it difficult to use fine ion exchange resins. It has been proven that softening of membrane filtered clarified juice is quite efficient. However, the cost of membrane filtration remains relatively high. A fractal softener with a short bed has been tested for unfiltered clarified juice. Test results and the benefits of clarified juice softening are discussed.

Introduction

Softening of sugar juices and molasses has become an established unit operation in the beet sugar industry, especially in factories using chromatographic technology for molasses desugarization. Because of the negative effect of divalent cations on separation characteristics of chromatographic resin, juice or molasses softening is a necessary part of the process. It has been noted that processing soft juices and syrups positively affects the downstream operations. It eliminates the need for descaling evaporators and improves heat transfer efficiency. The softening process also improves boiling characteristics of juice and has a positive effect on sugar quality. Several ion exchange processes have been developed and implemented over the years. Reviews of these methods can be found elsewhere (Dorfner (1991), Van der Pool (1998)). The cane industry so far has not taken advantage of the softening process.

Although much attention has been paid by various researchers to resin regeneration and regenerant waste handling, relatively little has been done for development of more efficient equipment. Conventional ion exchange comprises regular tanks with relatively simple fluid distribution systems. Typically larger diameter tanks are less efficient because of fluid maldistribution. These inefficiencies are often remedied by increasing resin bed depth, which leads to underutilization of ion exchange capacity, excessive regenerant use and unnecessary dilution of process streams. More advanced systems, such as Calgon Carbon's ISEP (Snyder (1999)) use countercurrent carousel design, allowing exhausting resin beds more completely. They also use smaller process vessels that reduce problems with fluid distribution.

New opportunities for design of ion exchange equipment arise with the implementation of engineered fractals for fluid distributors (Kearney (2000), Kochergin (2001)). By providing nearly ideal fluid distribution, fractals allow utilization of very shallow resin bed and therefore, significantly reduce required amount of resin. Kearney (2001) has presented data that demonstrated advantages of using fractal distributors for softening of beet thin juice. Table 1 illustrates the differences in softening systems utilizing conventional and fractal softeners. The new equipment is about ten times smaller compared to conventional systems due to more efficient distribution and the possibility of faster cycling. Smaller pressure drop allows building cheaper equipment and saving energy.

Table 1

Comparison of conventional weak cation juice softening system with an industrial flatbed fractal device (Amalgamated Sugar Company, Paul factory, Idaho, USA)

Parameter	Conventional (lateral orifice distributor)	Fractal Flat Bed
Resin bed depth, m	1.0	0.15
Exhaustion flow rate, BV/hr	50	500
Bed pressure drop, bar	3.5-5.6	0.1
Regeneration flowrate, BV/hr	30	150

Softening in cane mills has not been considered as a standalone operation, mainly because of the presence of suspended solids in clarified juice. Because of the possibility of plugging the resin bed with suspended solids, clarified juice has to be filtered prior to ion exchange. Suspended solids level in beet thin juice (about 5 ppm) is much lower compared to cane clarified juice (up to 200 ppm). Higher suspended solids level in cane juice makes it difficult to use fine ion exchange resins because of the danger of plugging. Cane juice typically also has higher hardness compared to beet thin juice, which means that larger softening systems could be required and more regenerant will be produced. Therefore, softening of clarified juice is typically considered in combination with other separation methods developed, for example, for white sugar production directly in the cane mills, (Kochergin, e.a. 2000) (Fechter,e.a. 2001). It has been proven in many publications that micro- or ultrafiltration provides the juice of sufficient quality to avoid plugging of ion-exchange beds. Although membrane filtration positively affects cane mill operation, the cost of membrane systems is still relatively high to justify the required investment. Apparently if the softening process could be carried out without prior membrane filtration and the overall softener size and cost could be reduced, the process would be of interest to cane processors worldwide. A fractal softener with a short bed has been tested for filtered and unfiltered clarified juice. Test results and the benefits of clarified juice softening are discussed below.

Experimental section

Tests have been performed with membrane filtered and unfiltered cane clarified juice. A pilot fractal softener (3.24 sq. ft. cross section, resin bed height – 6- 10 inches) was installed at the Sugar Cane Growers Cooperative of Florida mill (see figure 1). Clarified juice was prescreened through a Filtomat screen with a 100 micrometers slot size. The first set of tests was carried out using clarified juice filtered through a Koch UF membrane (MWCO around 100,000). A DOWEX strong cation resin in Na+ form was used for softening. Flowrate was maintained at about 55-60 BV/hour. Resin regeneration was performed using NaCl solution –1.78 eq/l Na – up to 180 % on operating capacity to fully regenerate resin. Over a hundred exhaustion - regeneration cycles were performed. Average feed brix- 11.7%, feed limesalts (determined by EDTA titration) - about 22.5 meq/100g DS. Average service cycle was about 70 minutes. Typical steps involved in a full production cycle are listed in Table 2. The sequence of steps and flow direction may vary depending on the selected process configuration.

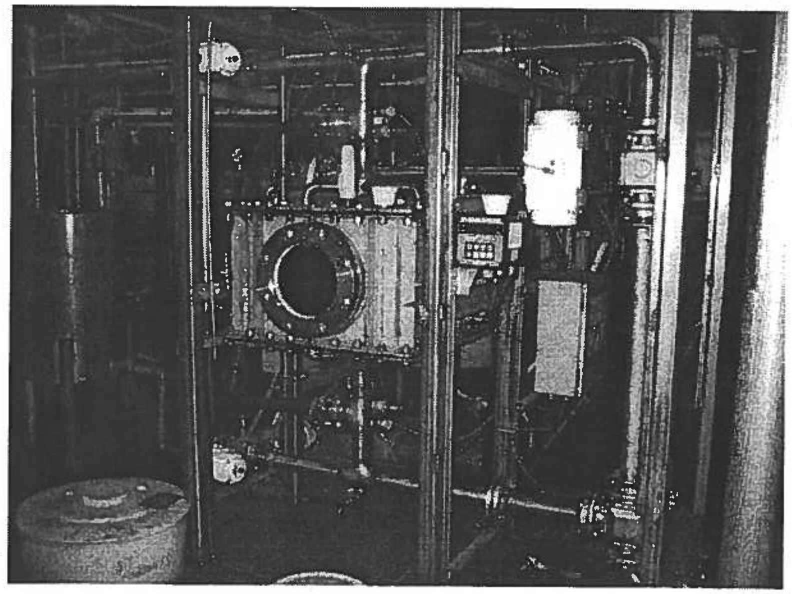


Figure 1 Pilot Fractal softener

Table 2
Typical softening steps

Step	Effluent stream composition	Comments (where to send the stream)
Sweeten on	Displaced water	To sweet water tank
Exhaustion (softening)	Soft juice	To mill evaporators
Sweeten-off	Dilute soft juice	To softener supply tank or mill evaporators
Resin backwash	Sweet water and suspended solids	To drum filters
Regeneration	Mixed chlorides (for strong acid in Na-form) Acidic Ca, Mg sulfate waste (for weak H+form)	Waste stream
Regenerant rinse	Dilute regenerant	Regenerant supply tank

Nine tests were run using unfiltered clarified juice to observe if the softener could operate without membrane filtration. The bed height was reduced to 6 inches to allow full resin expansion during the backwash stage. Juice flowrate was reduced accordingly. Pressure drop was monitored across the resin bed as well as the top and bottom fractals to determine if any irreversible plugging took place. Juice hardness was analyzed periodically by EDTA titration method to determine the breakthrough point.

Results and discussion

The results for seven tests with unfiltered clarified juice are summarized in Figures 2 through Figure 4. Increase in pressure drop was plotted against time of the softening cycle for top and bottom fractals and resin, respectively. Increase in pressure drop gives indication on plugging of fractal channels or resin bed with solids. Figure 2 illustrates that pressure drop increase was relatively small during the cycle in all but one case. Higher-pressure drop increase in that case was related to disruption in clarifier performance, which led to increased level of suspended solids in the feed material. The same trend can be observed for the resin bed, where pressure drop increase in test 4 was sharper then in the other tests. However, the pressure drop in the bottom fractal was not following the same trend indicating that the solids must have been held by resin and did not have the time to reach the bottom fractal within one operating cycle. Overall the values for pressure drop in the bottom fractal have not exceeded 0.35 psi. The fact that pressure drop was returning to the original value every time after backwash indicates that no irreversible resin or fractal fouling took place.

It is important to mention that the operating capacity of 1.7-1.8 eq/l was reached consistently throughout the tests. The same resin capacity was reached with membrane-filtered juice. The key conclusion is that a certain accumulation of suspended solids in the resin bed did not interfere with the softening process. Figure 5 shows a typical softening breakthrough curve for unfiltered juice also showing certain pressure drop increase during the operation cycle.

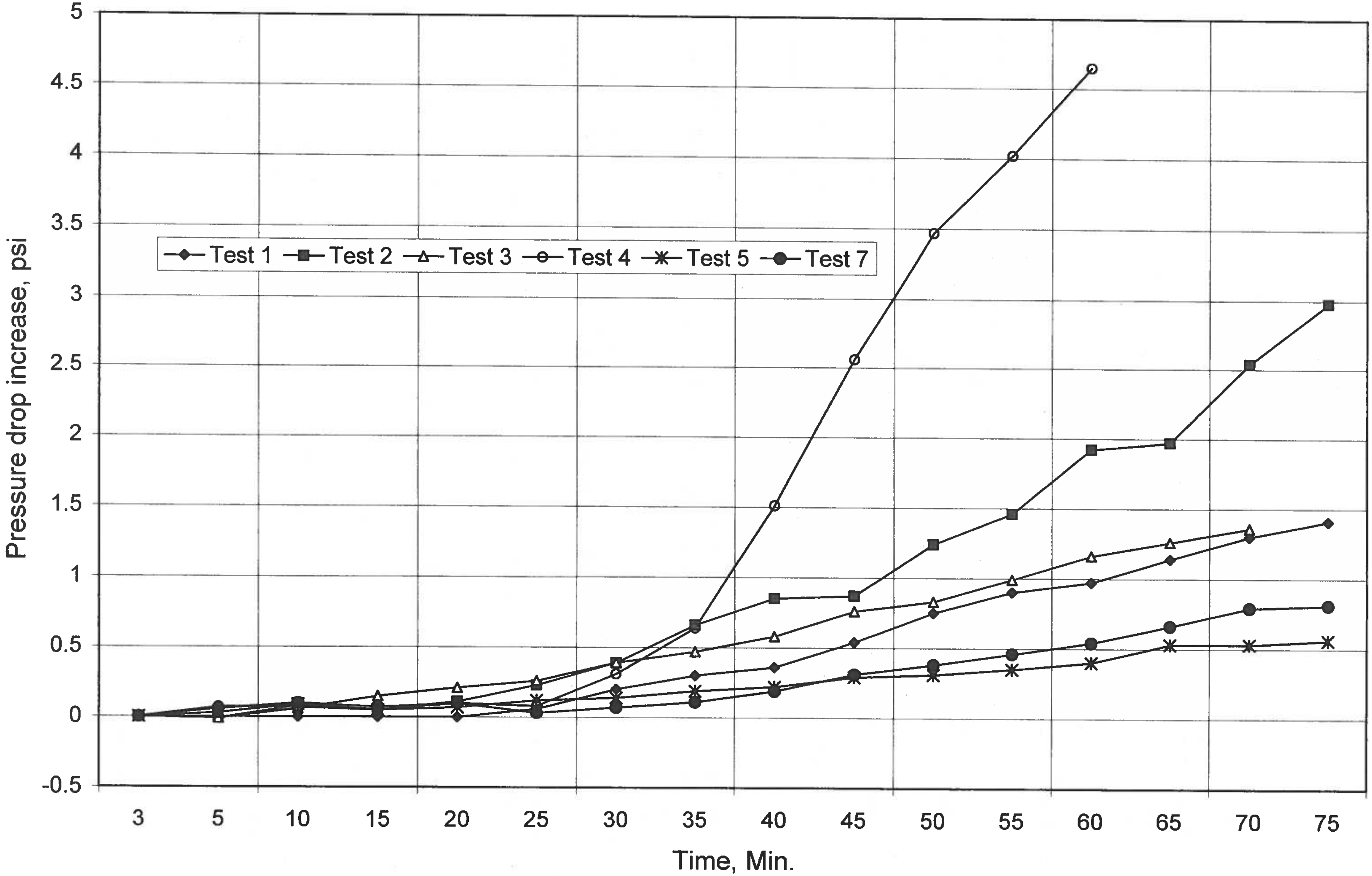
After the end of the test program it was discovered that the resin still contained suspended particles, which was due to insufficient backwash pump capacity.

Benefits of clarified juice softening

A short summary of expected benefits if clarified juice softening is implemented in a cane mill juice is shown in Table 3. The following discussion explains the potential advantages of juice softening.

Experience with beet sugar factories employing the juice softening technology indicates that no evaporator boilouts are required. This results in savings in chemicals, labor, repair and maintenance cost associated with boilouts. Because fully softened juice may become corrosive, a decision has to be made about the required level of softening. The

Figure 2 - Unfiltered Clarified Juice Tests
Top Fractal



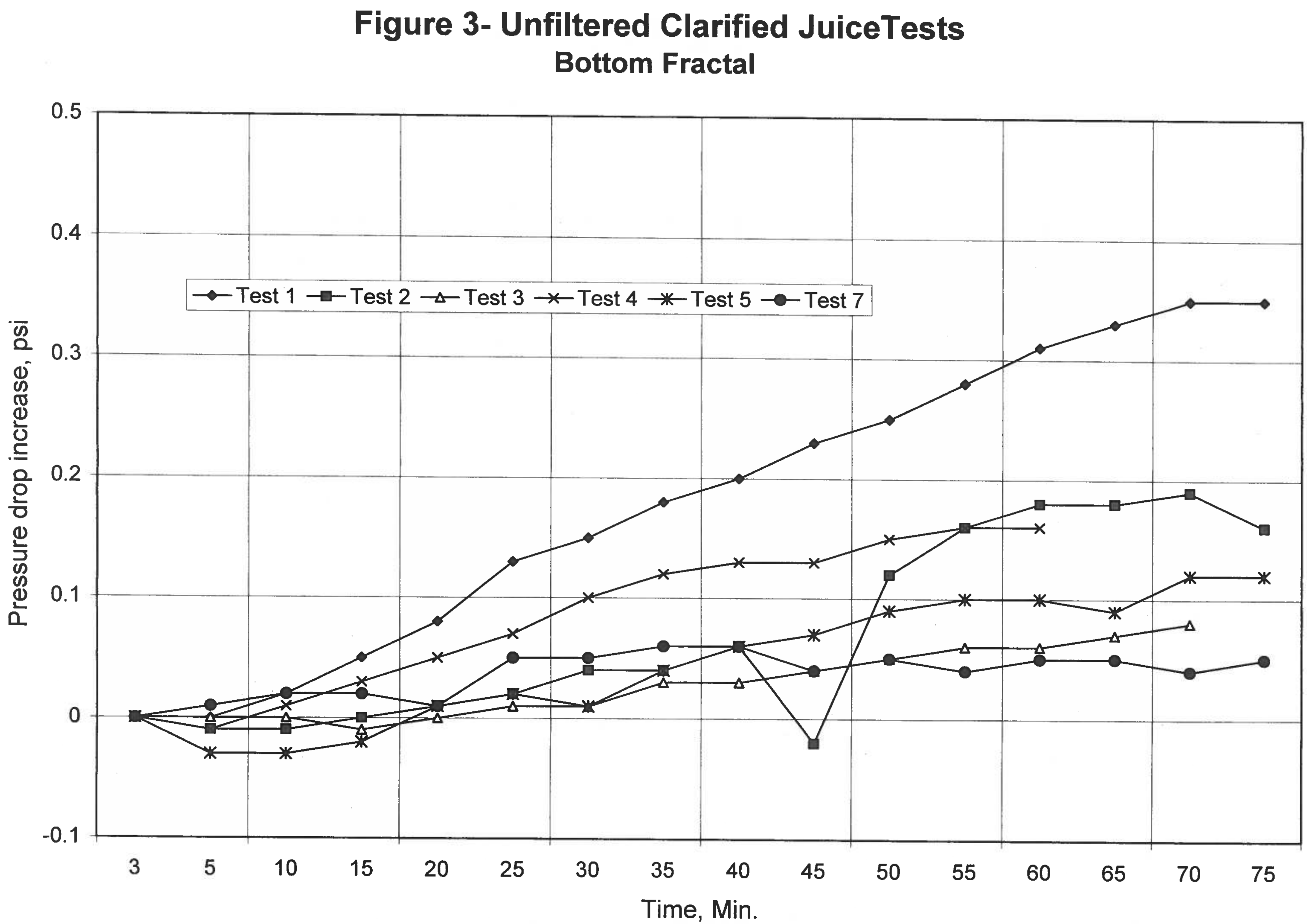


Figure 3- Unfiltered Clarified JuiceTests
Bottom Fractal
Test 1
Test 2
Test 3
Test 4
Test 5
Test 7
Pressure drop increase, psi
0.5
0.4
0.3
0.2
0.1
0
-0.1
3
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
Time, Min.

Figure 4- Unfiltered Clarified Juice Tests
Resin pressure drop

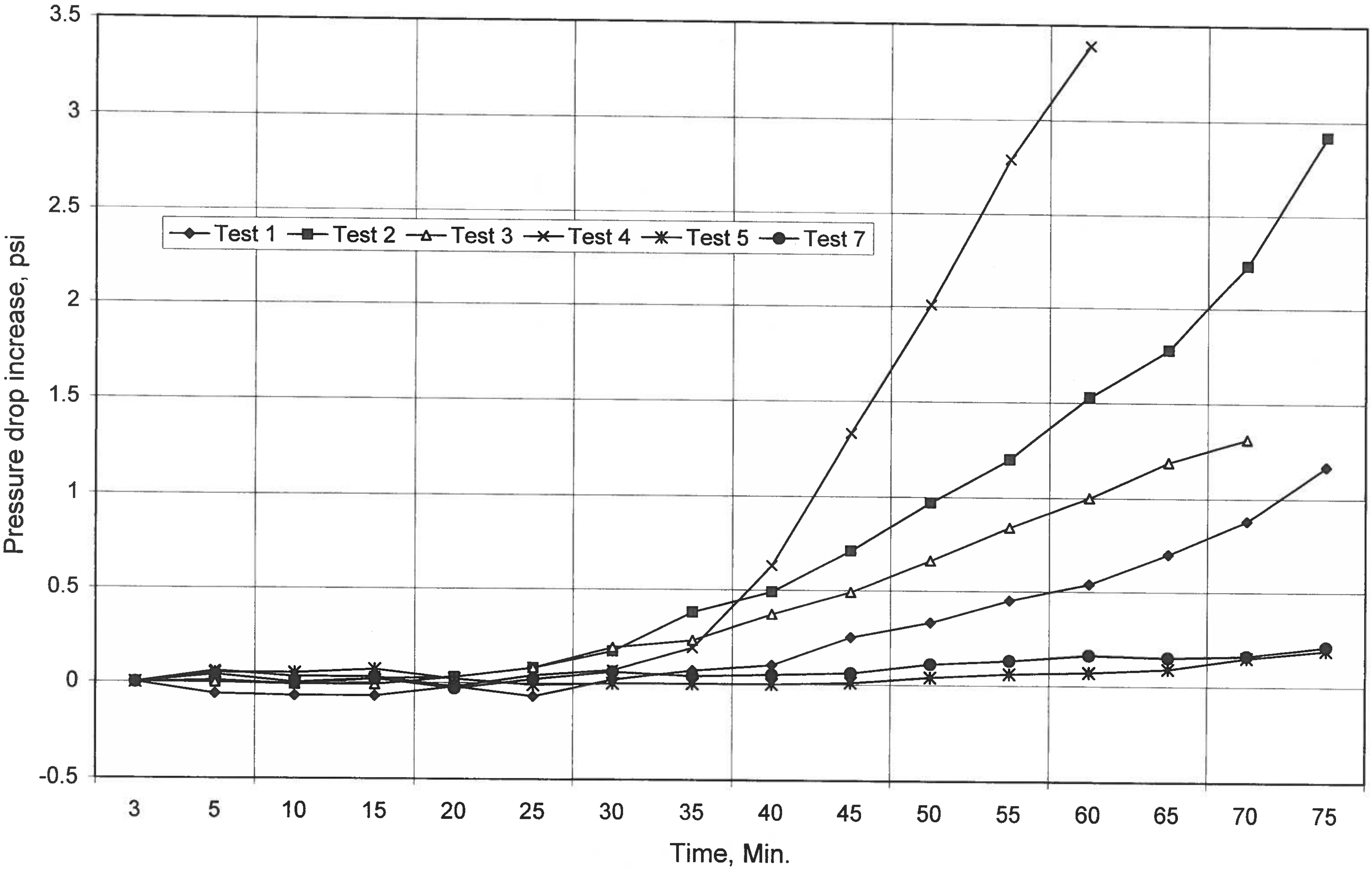
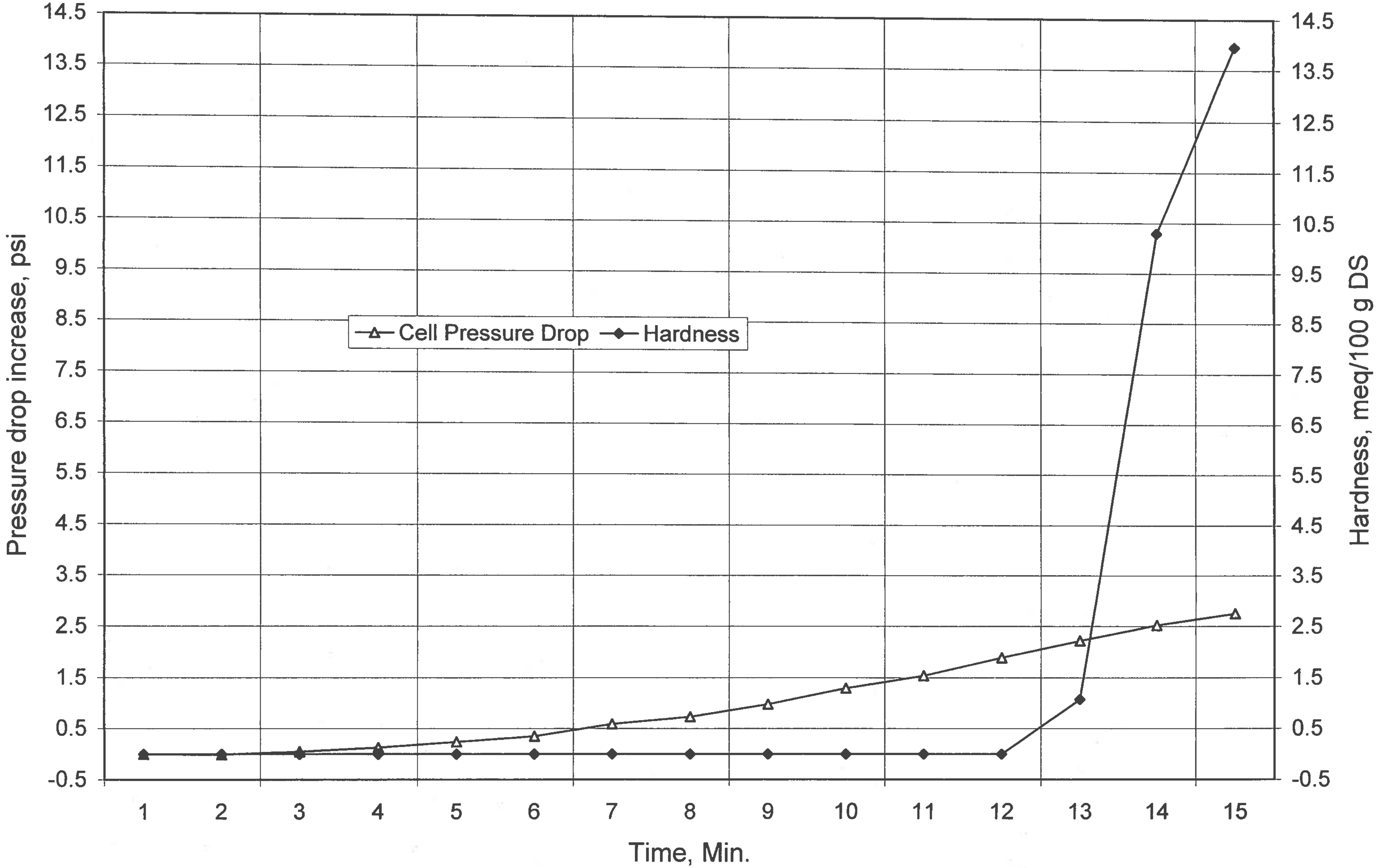


Figure 5 - Unfiltered Clarified Juice Tests

Typical breakthrough curve



residual concentration of calcium in the juice can be adjusted by extending the softening cycle and if necessary allowing a certain amount calcium to leak through the softener.

Gradual evaporator scaling during the period between the boilouts reduces overall heat transfer efficiency. In the case of softened juice the heat transfer coefficients stay constant, thus maximizing evaporator performance. In addition, the evaporator capacity unused during boilouts becomes available, when boilouts are eliminated. It may become an additional benefit for the factories where evaporation creates a bottleneck for capacity increase.

Potential increase in raw sugar quality deserves a separate discussion. The benefits, however, are hard to substantiate without running a full-scale test program. Clearly, elimination of calcium ions that tend to form insoluble salts should benefit the crystallization process downstream and improve the quality of final product. At the same time elimination of scale on the heating surface in evaporators and vacuum-pans will possibly allow reducing the temperature of heating steam and potentially alleviate color buildup. These benefits cannot be easily proven on the pilot-scale. Therefore, large-scale tests are recommended.

Table 3
Softening benefits summary

Benefits	**Comments**
Eliminate evaporator boilout	No calcium salts in the solution
Reduce chemical cost	Some chemicals will still be used for softening regeneration
Energy savings	See the discussion above
Labor and repair savings	
Potential for capacity increase	Availability of additional evaporation area
Potential improvement in raw sugar quality	Difficult to substantiate, needs to be tested on large scale
Potential color reduction	Lower temperature difference

Strong cation vs. weak cation softening

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Weak cation resins typically have higher operating capacity compared to strong cation resin (3.0-3.5 eq/l resin for cane juice), which would result in smaller size equipment. In the beet industry weak cation resins are used in H+ form. Regeneration is performed with dilute sulfuric acid. It had been proven for many years of industrial operation that the risk of sucrose inversion is negligible.

In the case of weak cation resins regenerant is used in excess of 10-20% over stoichiometric requirements. The main characteristic of weak cation softening is the production of large amounts of dilute regenerant. Acid dilution is required to prevent the formation of insoluble calcium salts (gypsum) in the resin matrix. In the beet industry thin juice softening is beneficial, because the waste liquid is used as a pressing aid for beet pulp (Henscheid (1990)). With a relatively low level of initial thin juice hardness (after carbonation) the total amount of softening regenerant is used in the process. There is no information in the literature, if addition of gypsum provides any advantages for cane milling. Also, with cane juice hardness being 2-3 times higher than that of the beet juice the total amount of dilute calcium sulfate solution will be proportionally higher.

Strong acid softening resins are typically used in Na-form and regenerated with about 2N brine solution. As a result regenerant is a rather concentrated solution of calcium, magnesium and sodium chlorides. The total operating exchange capacity of strong resin for cane juice reaches 1.7-1.8 eq/l of resin. The amount of regenerant typically exceeds the stoichiometric requirements by 50-80 %. The large amount of sodium in the waste stream may create an environmental issue for some locations. ARi has developed and tested on a bench scale a method allowing reduction in total waste by a factor of 10-15, which makes the application feasible. Another advantage of strong cation resin over weak cation resin for cane applications is its temperature stability, since clarified juice temperature is close to boiling point.

System size

Conventional softening systems are characterized by relatively large size and high resin bed making them essentially unsuitable for softening of unfiltered cane clarified juice. For example, a softening installation was described for processing of about 1000 gpm of membrane-filtered cane clarified juice, consisting of two columns, each containing 45 m^3 of strong cation exchange resin and 15 m^3 of inert materials (Kwok (1996)). The inert materials must be used to shield the liquid distributors from the resin. Each column is 7.6 m high and 4 m in diameter. Softeners had to be regenerated every 8-10 hours: 80 % hardness removal has been reported. It is obvious that the described installation would not be able to perform on unfiltered clarified juice. Because of the presence of suspended solids such installation can turn itself into a depth filter because of the long cycles and bed height.

Our estimates show that for fractal installations characterized by a short bed and fast cycling only about 3.4 m^3 for each unit would be necessary. No inert material is required. The accumulated solids will be removed during backwash cycle as was demonstrated in our tests. Short cycles mean that less solids will be "filtered" through the bed between backwash cycles. Shallow bed depth will assure the depth filtration is minimized and more fine solids are passed through without being held inside the bed.

Conclusions

1. Softening process can be accomplished on both filtered and unfiltered clarified juice. In the short bed fractal systems accumulation of suspended solids in the resin bed during the operation cycle did not result in reduction of resin capacity. Solids buildup is dependent on total concentration of suspended solids in the juice. A reliable prescreening will be essential to prevent solids from building up in the resin bed or distributors.

2. Use of fractal distributors in the softeners allows accomplishing the softening process using a shallow bed with a small amount of resin. Frequent cycling and backwashing allows avoiding significant buildup of solids in the resin bed. Pressure drop across the softening cells did not exceed 4 psi over the length of the operating cycle. Cell construction allows "out of cell" backwashing if it becomes necessary.

3. A decision on the type of softening resin depends on the environmental regulations and process configuration of a particular plant. A method of regenerant reuse for strong cation resin has been developed.

4. Longer large scale testing is recommended to verify that the softening system can withstand normal process fluctuations.

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