Thin Juice Deliming With Gel Type Cation Exchange Resins

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In 1964, with the objective of getting the best possible use from an evaporator installation which was inadequate in capacity, a thin juice deliming plant was purchased from the Dutch firm Imacti Industrieele Maatschappij Activit N.V. and installed.

In anticipation of a future increase in plant capacity, the service conditions for which the plant was designed were set at 22,000 Imperial gallons of thin juice per hour, with an average lime content of .075 CaO/100 Brix or approximately 15 grains $CaCO_3$ per Imperial gallon. Since the resin chosen, Imac C-12, had an estimated exchange capacity of 20,000 grains $CaCO_3$ per cubic foot/hour, it was estimated that a column containing 176 cu ft would remain on stream 11.4 hours, thereby requiring 2.1/10th regenerations per 24 hours, under average conditions. Therefore, the primary part of the installation consisted of two columns, each containing 176 cu ft of Imac C-12 resin operating in the sodium cycle and using common salt for regeneration.

Imac C-12 is a strongly acidic cation exchange resin obtained by sulphonating the gel type copolymer divinylbenzene; it can be used at temperatures as high as 120°C.

In addition to the two resin columns, the remainder of the installation consisted of a sweetening-off tank, having a capacity of two bed volumes; a buffer-tank for regeneration water storage of approximately four bed volumes; a decantation tank for resin recovery, having a capacity of one bed volume; a concrete wet salt storage tank, having an effective capacity of approximately 40 tons of salt. Also included were the necessary valves, piping and flowraters. A brine filter which was not part of the original installation was added later. All internal surfaces of the entire installation were epoxy-coated.

The material put through the columns was filtered second carbonation juice without sulphitation. Sulphitation of thin juice was discontinued to avoid sulphating of the resin and to put the juice into the evaporators at a pH high enough to avoid inversion. Recently, considerable attention has been given to the effect of temperature on dissociation constants. From Figure 1 (from data provided by S. Stachenko, Canada and Dominion

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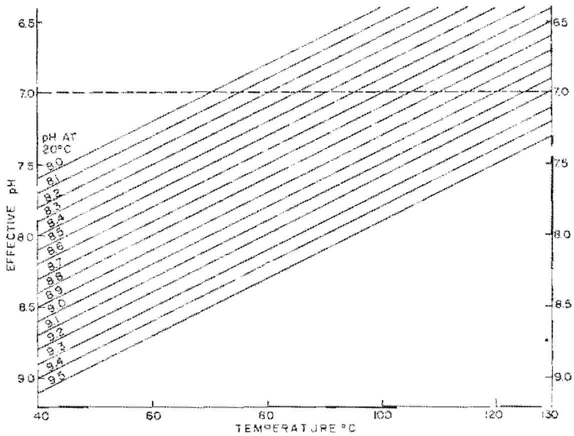

Figure 1.—Temperature effect on pH of thin juice.

Sugar Co. Ltd.), it will be seen that a pH of 9.2 at 20°C is actually an effective pH of 7.0 at 130°C. There was no decrease in pH due to lime removal.

The juice was put into the column at approximately 45 psi. pressure, estimated pressure drop across the column with a flow rate of 90 to 120 gallons per cu ft of resin per hour being 20 to 30 psi. It was planned to put the entire juice production through the resin until the effluent from the bed reached a lime content of 0.03 CaO/100 Brix after which the saturated bed would be sweetened-off and regenerated. It was estimated that a volume of sweetening-off water amounting to approximately twice the bed volume would be required and that the sweetening-off operation could be accomplished in approximately 20 minutes. Actually, the resin is sweetened-off to a solid content of 1° Brix as determined by small hand refractometer.

The sweet water is introduced into the evaporator supply tank through a control valve, operated on the split-range principle by the evaporator rate controller, in such a manner that no water can be added to the evaporator supply tank as long as sweet water is available. The pressure of the sweetening-off water is approximately 50 psi.

After sweetening-off, it is necessary to put the resin in suspension for efficient backwashing and reclassification of the bed. This is accomplished by an air purge requiring approximately 300 cu ft of air at a pressure of 7 psig supplied during a three-minute interval.

Following the air purge, the bed is vigorously backwashed counter-current to the juice flow until the effluent water is clean and free from suspended matter, including resin fines. This water is sent to discard through the decantation tank which retains the suspended resin and holds it for eventual recovery. After backwashing, the bed is regenerated with brine made from common crushed rock salt which is dissolved in the wet salt storage tank from which it is removed at 24° Bé, filtered and diluted to 12° Bé before being put through the resin in a counter-current manner.

The theoretical amount of sodium chloride required is 1,984 pounds per regeneration, which in practical operation amounts to approximately 2,000 pounds of common rock salt, or about 800 gallons of saturated brine. The resin is normally in contact with the brine for approximately 1 hour per regeneration.

In the 1967 campaign, a polyphosphate material known as Watcon 116 was introduced with the brine during every fourth or fifth regeneration, at the rate of 1 pint per 100 gallons of 24° Bé brine to assist in keeping the beds clean. After the brine treatment, the excess of salt is removed by counter-current back-washing. When the resin is free of salt, the column is again ready for another cycle. However, when the original installation was put into operation, certain unanticipated troubles developed.

The first difficulty concerned the method of brine preparation and withdrawal from the salt storage tank. As originally constructed, the solution water was introduced about midway in the salt bed, vertically, and the brine withdrawn from perforated transite pipes at the bottom of the tank. This resulted in plugging of the brine pump and lines. A revision was made, on advice from the Imacti Engineers, which involved constructing a barrier near one end of the tank, extending about halfway up the vertical height. This created a brine well to which the pump suction was connected through a filter. The solution water was then introduced at the bottom of the tank through the original withdrawal pipes. The brine overflowed the newly constructed barrier into the brine well from which it was pumped. No further difficulty from this source was experienced.

Secondly, it soon became apparent that the filtration of the second carbonation juice was completely inadequate. After a short time on stream, an excessive pressure drop across the column began to develop, reducing the column capacity and requiring the bypassing of more and more juice. This indicated the need for a trap filtration which was set up; at times, the results of the two filtrations were not always perfect.

Another difficulty was the occlusion of air in the juice, due to the fact that the juice flowed directly from the filter receiving boxes to the pump. This difficulty was eliminated by the installation of a surge tank between the filters and the resin columns, equipped with automatic level control.

A serious difficulty was traced to the fact that the brine was not being filtered. The salt purchased for regeneration has an insoluble content of .25% which is principally shale. When the difficulty became extreme, the columns were unloaded down to the supporting mat upon which was found a hard layer of shale, as much as 3″ thick, the only juice flow occurring through an annular ring about 4″ wide around the outer periphery of the column. This difficulty was eliminated by the installation of an Immedium brine filter.

After a considerable period of time, it was discovered, during a visit of one of the Imacti Engineers, that both the air for purging the columns and the water for backwashing them, was being supplied in insufficient volume. Inadequate air purge and backwash of the columns results in channelling, giving capricious and completely unsatisfactory results during both the regeneration and service cycles. On each regeneration, the resin must be put in suspension by the air purge and kept there by the backwash during the entire operation which results in some carry-over of resin into the decantation tank. There was a tendency on the part of the operators to limit the amount of carry-over by insufficient air purge and backwash because the resin from the decantation tank had to be recovered by a manual screening operation which turned out to be laborious and time consuming. To solve this problem, a small installation was set up for mechanically screening the resin and returning it to the columns, using a jet pump, pumping through a rubber hose to reduce damage to the resin to a minimum. This installation is shown in Figure 2.

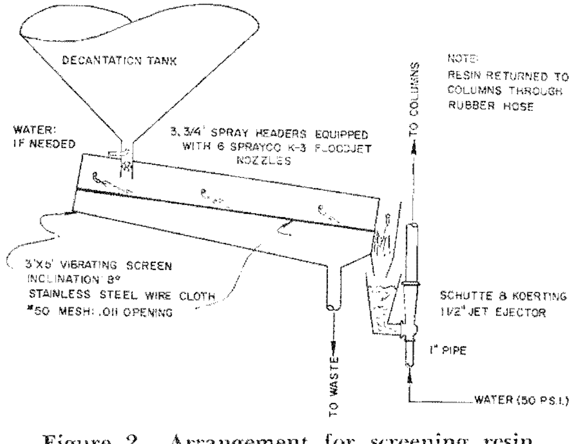

Figure 2.—Arrangement for screening resin.

After solving, or partially solving, the various difficulties, the deliming plant proved to be a valuable addition to the process equipment.

Although some of the various ramifications pertaining to the operation of the deliming plant, during the 4 years it has been in service, might be of interest, the scope of this paper does not permit their discussion. However, one important circumstance should be mentioned. It has never been possible to pass all of the thin juice through the resin columns.

During the 1967 campaign, the effluent from the columns had an average lime salt content of .019 while the lime salt content of the thin juice entering the evaporators was .035. Figure 3 indicates graphically that only 63% of the total juice was passed through the columns. This can be accounted for by one or more of the three following factors:

a) Filtration of the second carbonation juice is still inadequate.
b) The low raw melt is returned to the beet end causing a considerable increase in viscosity.
c) The estimated flow rate through the resin was too high and has not been realized.

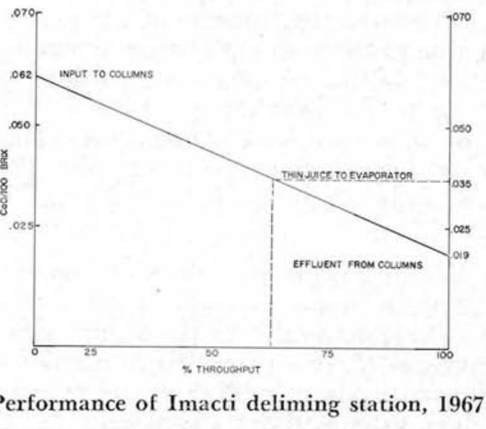

Figure 3.—Performance of Imacti deliming station, 1967 campaign.

Tables 1 and 2 show a very condensed comparison between certain results obtained in the 1963 campaign, which was the last operation without the deliming plant, and the 1967 campaign. As between the two campaigns, there was a 14% increase in slicing capacity. Lime salts decreased by 50%. The brix of the evaporator thick juice increased by 8%. Evaporator boil-outs decreased by 73%. Steam requirements, percent on beets, decreased by 12.2%. Much of the improvement indicated above can be credited to the deliming station.

Table 1.—Comparative data.

	Without deliming 1963[a]	With deliming 1967
Average daily slice	1341	1528
Lime salts, thin juice	.073/100 Brix	.035
Brix, evaporator thick juice	56.6	61.1
Evaporator boil-outs, Effects/100,000 tons[b]	8.0	2.18
Steam, percent on beets	97.75	85.89
Soda ash, lbs/ton beets	.93	Nil

[a] Last year of operation without deliming.
[b] Individual effects in five-effect system.

Table 2.—Cost of process materials for deliming.

	1967
Cost of salt/ton beets	\$ .0360
Cost of polyphosphates/ton beets	.0057
Cost of replacement resin/ton beets[a]	.0046
Total cost per ton beets	\$ .0463

[a] Replacement amounted to 7% of resin in service.

Since the deliming plant has been placed in operation, soda ash has been introduced into process in significant amounts only in 1965 when, due to the working of a large tonnage of extremely badly deteriorated beets, the effective alkalinity dropped as low as −.1311. Prior to the installation of the deliming plant, the introduction of soda ash began when the effective alkalinity reached .005 or lower. This procedure was discontinued and soda was added only when the pH of the low raw massecuite dropped below 7.5.

After the deliming plant was placed in operation, there was a noticeable decrease in the viscosity of the sugar end products. This was particularly noticeable in the fluidity of the massecuites, and had a beneficial effect on centrifugal and crystallizers operations. At the centrifugals, even high purity massecuites discharge from the loading gates without chunking. At the crystallizers, higher densities can be maintained without danger of damage to the stirrer mechanisms.

Conclusion

A deliming station has been described, the operation of which has resulted in reduced evaporator boil-outs, increased hydrogen ion stability and reduced viscosity of sugar end products. Solutions of some difficulties encountered were also described.