Full-Scale Ion Exchange Operations at the Hardin Plant of the Holly Sugar Corporation

E. A. HAAGENSEN[1]

A FULL-SCALE ion exchange plant was installed at the Hardin, Montana, factory of the Holly Sugar Corporation in 1947. The plan was to have the unit ready for operation at the start of campaign. However, delays in construction occurred and the ion exchange did not go into operation until November 27 and, because of minor adjustments, the plant did not operate continuously until November 30. The campaign finished December 21. During this period, all the juice from 36,550 tons of beets was processed by ion exchange. The results have not been completely analyzed and evaluated at this time, but a preliminary report on this question is deemed worthwhile.

Plant Description

The plant consists of four pairs of cells, each pair being one cation exchange cell and one anion exchange cell. All cells are identical in size and mechanical design. They are 10 feet in diameter by 10 feet straight side height with ASME spun heads, and are designed for 60 pounds per square inch working pressure. Rubber lining is used throughout and suitable underdrain, feed and overflow distributors of chemical resistant materials are installed in each. Each cell contains 410 cubic feet of the necessary resin (cation or anion), giving a bed depth of 5 feet, 3 inches.

All piping in contact with sugar juices or regenerant liquids is rubber lined. The valves used are Saunders type with a rubber diaphragm and are completely rubber lined when installed in rubber-lined piping. These valves are operated by air pistons controlled by solenoid pilot valves which are in turn controlled by switches and relays on the control panel.

The control panel which is 20 feet by 8 feet by 3 feet, 6 inches, is dust and moisture proof and contains the controlling relays and instruments. On the face of the panel are mounted the instrument dials and the numerous control switches. The switches for operating the valves are grouped in five sets. One set is for the valves common to all cells and each of the other sets applies to the valves for each pair of cells. These switches have three positions, viz:—off, manual and automatic. When in the automatic position, various steps in the process involving several valves and pumps are controlled by a series of push buttons, thus minimizing errors involved in the human element.

The instruments consist of flow indicators on acid, ammonia, sweet water, cation and anion backwash, rinse water and reclaimed water; flow indicators and recorders in ingoing and outgoing juice; recording and indicating pH meters on cation and anion effluent; and conductivity indicators and recorders on untreated juice, cation and anion effluent, sweet water and rinse water.

The regeneration of the cation cell is three-stage, using 5 to 6 percent sulfuric acid. On the anion cells it is single stage, using 2 percent ammonia.

The juice to be treated is cooled by passage through spiral type heat exchangers on the other side of which is the outgoing treated juice from ion exchange, which is warmed up at the same time. Since this cooling is not enough, the incoming juice, after passage through the heat exchangers, is further cooled in a tubular cooler by means of factory supply water.

In addition, there are the necessary accessory equipment such as pumps, air compressors, tanks and storage facilities for the process.

The equipment is housed in an addition to the factory as a separate unit yet an integral part of the factory. It is so designed that normal operation of the factory may be resumed merely by opening and closing three valves.

Process

The process used was that involving treatment of second-carbonation juice. Filtered second-carbonation juice was cooled to below 20 degrees Centigrade to minimize inversion in the subsequent passage through the acid conditions of ion exchange. After cooling, the juice was pumped through one pair of cells; it first passed through the cation cell and then through the anion cell. This flow was continued until one of the pH meters or conductivity meters indicated that break-through had been reached. This indication was a rise in pH of cation effluent or a fall in its conductivity, or a fall in pH of the anion effluent or a rise in its conductivity. Any one or more of these indications was used as a criterion for determination of the end of a treatment cycle.

A detailed explanation of the process is not presented here, since various phases of the actual steps taken are rather well known and have been in use some time in small-scale units. However, it will be well to explain the decision to operate the ion exchange on second-carbonation juice. The treatment of raw juice is desirable from the viewpoint of elimination of lime and the accompanying kiln, carbonation, thickeners and filters. However, the process of treating raw juice still presents problems that have not been economically solved. In addition, the exchangeable ion load per ton of beets is 25 to 35 percent higher for raw juice than for filtered second-carbonation juice. Second-carbonation juice was chosen instead of sulfured thin juice because of the possibility of eliminating the purchase of sulfur. The desirability of this is not definite. It is known that oxidizing agents or substances are harmful to the anion exchange resins. Such oxidizing agents might be reduced by the use of sulfur dioxide and rendered harmless to the anion resin. This is a question that will require further study.

Results Obtained

The results during the brief operation at the end of campaign are probably not a true picture of ion exchange, since they were obtained when treating beets that had been in storage for some time. The beets were in relatively poor condition a good deal of the time. Soft and sour beets were common and many were black and dried out. Table 1 shows the average results for a day's operation.

Table 1.

	Feed 2nd-carbonization juice	De-ionized juice
Brix	12.5	9.6
Purity	89.6	97.6
Percent sugar	11.0	9.5
Conductivity mmhos	4325	245
Nigrogen*	126	55
pH	9.2	7.5
Percent dilution		15.8

*An arbitrary nitrogen coefficient.

From table 1 it may be seen that, based upon purities, there is a non-sugar removal of 82.5 percent. The nitrogen removal is 56 percent. This is total nitrogen and is an indication of the organic removal. No determinations were made on ash removal, however, the conductivities may be taken as an approximation and indicate a 94 percent elimination. The percentage dilution is calculated on sugar content. The remaining loss in brix is due to removal of non-sugars. The dilution varied from 13 to 16 percent throughout the period.

Graph No. 2 shows the brix of the affluent of a pair of cells during sweetening-on plotted against effluent flow. The flow is expressed as percent of the total resin volume in both exchanger beds. The indication of brix in the effluent up to about 55 percent of the flow is not well accounted for. If it is presumed that there is some short circuiting of the incoming juice, it would be assumed that this brix should uniformly rise from zero. No tests were made on actual sugar present in this effluent by other means, but it is thought that this is not sugar but other substance. From the graph, it can then be seen that the brix is substantially zero up to 55 percent of bed volume, and then rises to full brix at 115 percent.

Graph No. 3 shows a similar plot of brix versus flow on the effluent during sweetening-off. Here again the effluent comes out at full brix of the solution in beds until about 55 percent flow and at 115 percent flow has about reached a minimum and is starting to level out. On sweetening-off, the purity of the effluent also drops. A good portion of the brix present in the effluent is due to non-sugars being displaced from the bed by the salts in the raw water used to sweeten off.

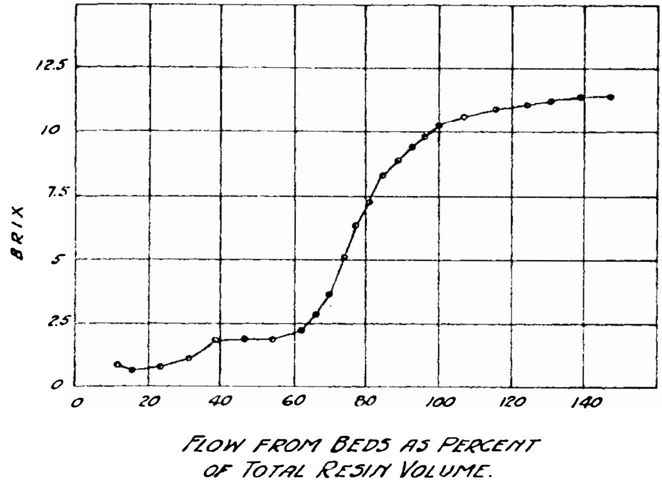

FIG. 1. - SWEETENING-ON

From a theoretical analysis of the resin beds, there is a total liquid content of a given volume resin of about 62 percent. The actual inter-granular voids are approximately 47 to 48 percent; the other 14 to 15 percent is the intra-granular voids or solution actually held in the gel structure of the resin. The inter-granular voids are displaced rather completely by incoming liquid, while the intra-granular voids are removed by a leaching action. The apparent discrepancy between 48 percent and the 55 percent on the graph is due to 1--the extra liquid in pipe lines and above the beds and 2--the fact that the displacement is not 100 percent efficient.

During the short period of operation, the quantity of chemicals used was varied quite widely. Good results were obtained with 25 pounds of 93-percent sulfuric acid and 6 pounds of anhydrous ammonia per ton of beets. It is believed that lower quantities of chemicals will be satisfactory, but time was not available to determine the minimum quantity practical.

Sugar-End Operation

The D-I thin juice was free from scale-forming substances and evaporation station performed very satisfactorily. In fact, the juice removed scale from heating surfaces and pipe lines. It, in all probability, dissolved some scale, but it also loosened scale which appeared in the pipe lines.

The juice darkened upon concentration in the evaporators. However, the color was still very light. The thick juice had a very bland taste and was free of the characteristic beet taste or odor.

There were problems encountered in the sugar boiling that, although forseen, were not as easily solved as would be expected. The high-purity juices with small percentages of invert were foreign to normal beet factory experience and, as a result, the art of the sugar boiler required adaptation to meet the demands of a new type juice.

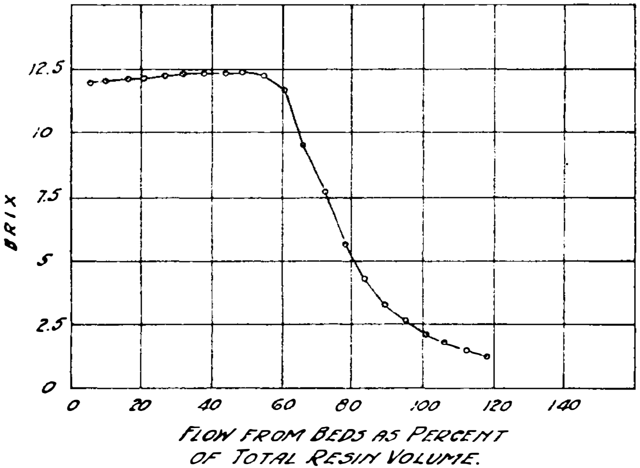

FIG. 2.-SWEETENING-OFF.

There were not any pronounced difficulties with the white sugar boiling. The pans grained well and the massecuites spun well in the centrifugals. The juice darkened considerably during boiling and molasses off the final pan was very dark.

During the period, it was not possible to produce a satisfactory white sugar off the second boiling of the green syrup from the first boiling. The sugar produced from this second boiling was much lower in ash than the regular run beet sugar, but it had a very slight yellowish color and was deemed unsuitable for marketing. It was remelted together with the other remelt sugars and reboiled.

There were some difficulties experienced with graining on the lower-purity pans. The increase in the rates of invert to sucrose was thought to partially account for this. The different viscosities of the juice effected the sugar boiler's judgment, which is based upon experience with normal beet juice.

A five-boiling system was attempted with the last pan of such a purity that it would yield 60-purity molasses. Part of the time this was realized. However, frequently, a pan did not yield as well as it should have, and the green purities from it were high. This entailed extra boiling, and a six-boiling system was used part of the time to correct this.

The lack of pan storage for the many liquors was a handicap in adhering to a boiling system. This is being studied for improvement of this phase in the future.

Sugar and Molasses Produced

The sugar produced was of superior quality. The ash content of the sugar was very low. By conductance measurements, it varied from 0.0001 to 0.0020 percent with an average for the period of 0.0008 percent. The SO_2 content averaged 0.3 ppm. There was a marked improvement in candy grade and a rise in darkening temperature during the candy test The sugar has a fine appearance and compares favorably or is superior to sugar normally produced in a milk of lime sugar factory.

The quality of molasses produced varied. This was probably due to inexperience with the new type syrups. The apparent purities sometimes rose appreciably above 60. The molasses had none of the characteristic odor or flavor of beet molasses. It did have a rather strong flavor characteristic of a very strong caramel or burnt-sugar taste. It was low in ash and high in invert, running from 8 to 16 percent invert on dry substance.

Conclusions

It is difficult to draw satisfactory conclusions because the run was short and because the beets, during the period of operation at the end of campaign, were storage beets and in relatively poor condition. All the data has not been analyzed and full results and conclusions are not yet drawn. However, it can be concluded that

1. Ion exchange produces a superior sugar.

2. Final molasses purities below 60 are obtainable with proper boiling techniques.

3. Ion exchange, on these particular beets, can be operated with a chemical consumption of 25 pounds of 93-percent sulfuric acid and 6 pounds of anhydrous ammonia per ton of beets to produce satisfactory results.