# Ion Exchange Process for Beet Sugar Refining
## II. Purification of Sugar Solutions

G. Assalini and G. Brandoli

*Received for publication April 29, 1960*

From an economic point of view, it would be of interest to the beet sugar industry to purify, between campaigns, by-products which are stable to storage. The exploitation of ion exchange installations to be used for this purpose, can be spread over 12 months instead of two or three months as for installations which treat beet juice only during the campaign.

A process has been studied which employs ion exchange resins for the purification of molasses inexpensively. During the sugar campaign, this process is able to treat, instead of molasses, the mother liquor of 75% purity which comes from the centrifugation of the first product which, in the European system, roughly resembles low-machine syrup in the U. S. beet industry. The mother liquor so treated has the same purity and density characteristics as a defecated thin juice before evaporation. Thus, during the campaign, the sugar factory could produce only first product raw sugar, eliminating most of the concentration, centrifugation, and conditioning stages of the low-grade products.

The so-called Assalini "B" Process involves only a partial removal of mineral salts and organic non-sugars from molasses by means of a rather novel ion exchange technique. A brief description of the operational method is as follows:

The molasses from the conventional process is diluted with an equal volume of certain fractions of the effluents from the "B" Process which are called "recycles." These recycles have a Brix of 10 or higher and a sugar content of 5 to 6%. This dilution is necessary both to decrease the viscosity of the molasses and facilitate its passage through the resin bed and to obtain a concentration of non-sugars sufficiently low to facilitate their separation from the sucrose in the ion exchange process. All of the solutions are at room temperature.

The installation consists of two columns and the flow pattern is shown in Figure 1. The first contains a porous anion exchanger such as Amberlite IRA-401S and the other a cationic resin such as Amberlite XE-200, a strongly acidic, porous cation exchanger in the 30-50 mesh particle size range. Both columns contain the same quantity of resin. Taking the resin volume of each column as 100, the following solutions are charged:

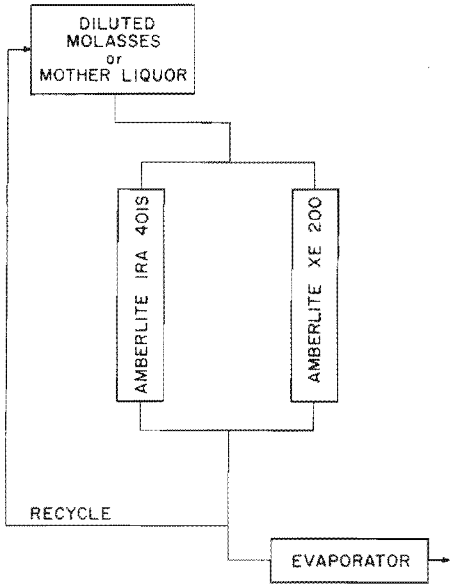


Figure 1.—Assalini "B" process.

*Amberlite IRA-401S Influents*

1. 20% resin volume of diluted molasses
2. 100% resin volume of water to displace the sucrose from the resin
3. 12% resin volume of 5% sodium hydroxide solution
4. 48% resin volume of water to wash the caustic from the resin

In all, for each cycle, there is charged a total volume equal to 180% of the resin volume.

*Amberlite XE-200 Influents*

1. 20% resin volume of the diluted molasses
2. 80% resin volume of water to displace sucrose from the resin
3. 12% resin volume of 10% sulfuric acid solution
4. 68% resin volume of water to wash the acid from the resin

Again, the charge of liquid equals 180% of the resin volume.

The effluents are taken off in different fractions as follows:

**Table 1.—Amberlite IRA-401S Effluents.**

| | |
|---|---|
| 1st Fraction: | 67% resin volume contains non-sugars and a little sucrose. |
| 2nd Fraction: | 10% resin volume which represents the recycle which is mixed with the cationic column recycle for the dilution of molasses for the next cycle. |
| 3rd Fraction: | 70% resin volume. This contains the purified sucrose liquor which, after mixing with the purified effluent from the cationic resin, goes to evaporation. |
| 4th Fraction: | 33% resin volume. This contains non-sugars, spent regenerants and a little sucrose. |

The average chemical analyses of these effluents are shown in Figure 2.

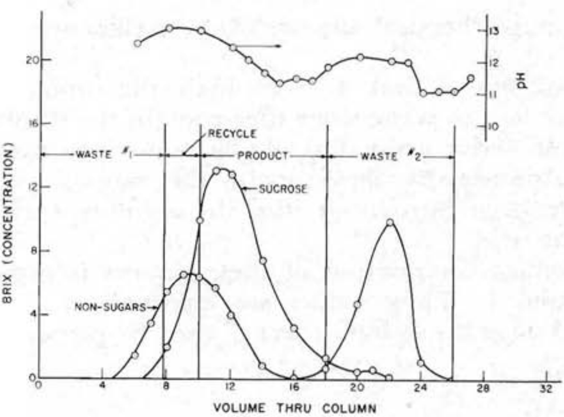


**Figure 2.—Typical effluent pattern for anion exchange bed, Assalini "B" Process.**

**Table 2.—Amberlite XE-200 Effluents.**

| | |
|---|---|
| 1st Fraction: | 40% resin volume containing a little non-sugar and traces of sucrose. |
| 2nd Fraction: | 44% resin volume representing the purified portion. This is mixed with Fraction 3 of the anionic effluent and is evaporated to produce refined sugar. |
| 3rd Fraction: | 10% resin volume. This is the recycle which, when mixed with Fraction 2 of the anionic column, is used for the dilution of the molasses. |
| 4th Fraction: | 86% resin volume containing non-sugars, spent regenerant, and a little sucrose. |

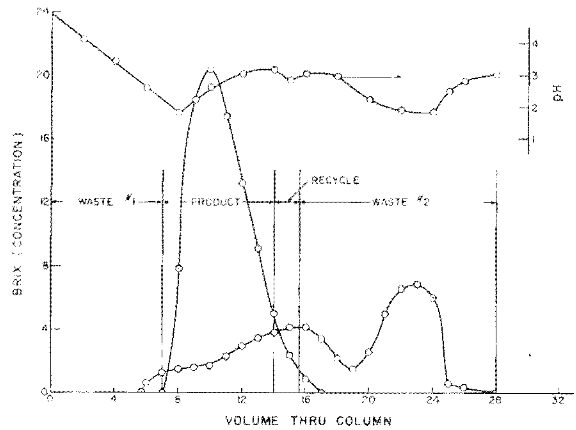


**Figure 3.—Typical effluent pattern for cation exchange bed, Assalini "B" process. Product fraction: upper curve—sucrose; lower curve—non-sugars.**

The average chemical analysis of these effluents are shown in Figure 3.

Fractions No. 1 and 4 from both the anion and cation columns are sent to waste since they contain the displaced water, little sucrose and a great deal of the non-sugars present in the molasses. Additionally, they contain the various spent reagents which have been introduced into the columns such as caustic and sulfuric acid.

The average composition of these liquors is summarized in Tables 3 and 4. These values are reported on a solids bases.

Table 3 gives the over-all effect of the "B" process on the final product.

**Table 3.—Average Analysis of Assalini "B" Process Liquors.**

| Component | Original Molasses | Thick Syrup | Evaporated Massecuite | Raw Sugar | Residual Molasses |
|---|---|---|---|---|---|
| Sucrose-polarimeter corrected | 51.00 | 47.14 | 73.52 | 89.57 | 53.13 |
| Sucrose—true | 52.24 | 45.91 | 72.66 | 87.77 | 51.44 |
| Percentage solids, refractivity | 82.11 | 59.88 | 92.79 | 97.47 | 86.41 |
| Percentage solids, true | 81.57 | 60.12 | 93.15 | 97.83 | 86.71 |
| Purity ratio, refractivity | 62.11 | 78.72 | 76.66 | 91.89 | 61.48 |
| Purity ratio, true | 64.04 | 76.36 | 78.00 | 89.72 | 59.32 |
| pH | 10.30 | 9.38 | 9.17 | 9.10 | 9.50 |
| Invert, % true sucrose | 0.019 | 0.414 | 0.276 | 0.051 | 0.916 |
| Total nitrogen, % true sucrose | 3.38 | | 1.47 | 0.44 | 3.72 |
| Total non-sugars, true value | 29.33 | 14.21 | 20.49 | 10.06 | 35.27 |
| Mineral non-sugars | 11.94 | . | 6.82 | 2.81 | 11.77 |
| Organic non-sugars | 17.39 | ...... | 13.67 | 7.25 | 23.70 |
| Organic non-sugars from nitrogen | 11.03 | ...... | 6.69 | 2.44 | 11.97 |
| Organic non-sugars, non-nitrogen | 6.36 | | 6.98 | 4.81 | 11.53 |

Table 4.—Average Analysis of Assalini "B" Process Liquors.

| Component | First Anion Fraction | First Cation Fraction | Fourth Anion Fraction | Fourth Cation Fraction |
|---|---|---|---|---|
| Sucrose—polarimeter | 0.33 | 0.52 | 1.29 | 0.35 |
| Solids—refractivity | 4.55 | 1.60 | 3.00 | 5.00 |
| pH | 12.0 | 2.80 | 12.0 | 1.90 |
| Invert, % true solids | trace | trace | trace | 0.37 |
| % nitrogen, % true solids | 3.18 | 4.06 | 2.80 | 3.48 |
| Total non-sugars | 4.22 | 1.08 | 1.71 | 4.65 |
| Mineral non-sugars | 1.77 | 0.20 | 0.70 | 2.12 |
| Organic non-sugars | 2.45 | 0.88 | 1.01 | 2.53 |
| Organic non-sugars, nitrogen | 0.91 | 0.41 | 0.52 | 1.09 |
| Organic non-sugars, non-nitrogen | 1.54 | 0.47 | 0.49 | 1.44 |

Fractions No. 3 and 2 respectively of the anion and cation resins when mixed together, represent the product portion which is sent to evaporation. Its average composition is as follows:

| | |
|---|---|
| Brix | 12.5-13.5 |
| Polarization | 10 -10.5 |
| Purity | 78 -80 |
| pH | 8 -10 |

The color (Stammer units—100% solids) is lower than that of the normal juice of the same purity.

The syrup obtained on evaporation of the product fractions corresponds approximately to the syrup which is obtained after centrifugation of the first product in European beet factories. Thus, the sugar which can be obtained is a normal second product sugar.

The mother liquor from the centrifugation of the massecuite is in turn a molasses which has the same characteristics as the ordinary final molasses in Europe. This is true since the organic and inorganic non-sugars are removed in approximately the same ratio as they occur in the original molasses. Consequently, the "secondary" molasses obtained from the "B" Process contains roughly the same ratio of impurities as the original molasses treated. It should be noted that this secondary molasses has a refractometric purity higher than the true purity which is the reverse of that usually observed for the molasses obtained in the conventional way.

The analytical data of the recycles are not included in the table since they are used to dilute the molasses for subsequent cycles.

The exact mechanism of the Assalini "B" Process is not known. However, it is apparent that the extremely low level of regenerations used, roughly one-sixth of that which would be normally employed in conventional deionization, results in a relatively small stratum of regenerated resin in the upper part of the column. It is possible that, as the regenerant ions are displaced from this portion of the column, successive ion exchange processes occur farther down in the bed. Ion exclusion plays a role in this process. Of interest to the mechanism of the process is is the fact that the void volume of the exchangers is approximately 40 percent.

The application of the "B" Process gives interesting economic results under European conditions where the Steffen Process is almost never used. Using a molasses with a purity of 60 and considering this as a limiting value for further crystallization, a purity of about 80 can be obtained in the purified liquor. In this case, it is possible to extract 50% of the sucrose originally present in the molasses. When processing mother liquor coming from the centrifugation of the first factory product, which has a purity of about 75, juices are obtained with a purity of about 90. This would represent about 28% more extractable sucrose as compared to that extractable with the conventional method without application of the "B" Process.

The operating expenses necessary to achieve these levels of purification are quite low. The materials consumed are as follows:

| | |
|---|---|
| Sulfuric acid (66 Be): | 0.099 lbs./lb. of sucrose in molasses treated |
| Sodium hydroxide 100%: | 0.05 lbs./lb. of sucrose in molasses treated |
| Fuel Oil: | 0.274 lbs./lb. of sucrose in molasses treated |
| Water: | 3 U.S. gallons/lb. of sucrose in molasses treated |

These consumptions are obviously very small and represent an economical method for extracting sugar from low-grade products.

The optimum conditions of operation using a typical molasses from an Italian beet factory, are indicated in the following table:

| | **Anionic Column** | **Cationic Column** |
|---|---|---|
| pH | 5 to 10 | 5 to 10 |
| Maximum temperature | — | 20° to 25° C. |
| Resin bed height | 160 cms. (5.25 ft.) | 160 cms. (5.25 ft.) |
| Time per cycle, minutes | 120′ | 120′ |
| Operating flow rate | 0.015 liters/liter resin/ min. (0.11 gpm/$ft^3$) | 0.015 liters/liter resin/ min. (0.11 gpm/$ft^3$) |

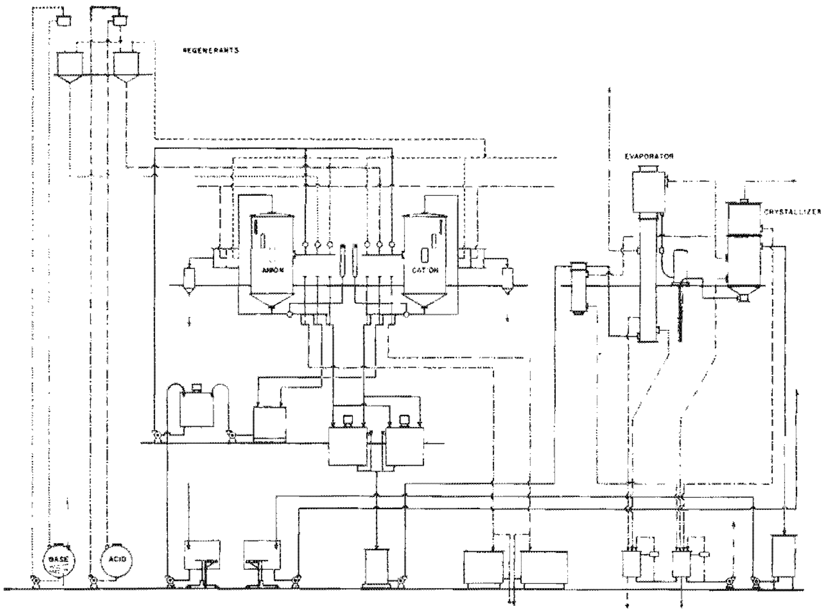


**Figure 4.—Flow diagram for an industrial plant. Heavy line indicates sucrose piping.**

**Table 5.—Income from Assalini "B" Process.**

| | |
|---|---|
| Sucrose in molasses charged | 15,300 tons |
| % extractable—54.61 | |
| *Total extractable sugar* | 8,355 tons |
| $\frac{8355}{0.7867}$ | 10,621 tons raw sugar to be refined |
| Refinery losses 0.80% of 10,621 ton = 85 tons | 10,536 tons (after losses) |
| The extractable is 10,536 tons × 0.7867 | 8,288 tons of 100% saccharose |
| $\frac{8288}{0.998}$ | 8,305 tons refined sugar |
| $\frac{8305}{0.9912}$ | 8,379 tons refined sugar-gross weight |
| 8,379 tons at $205.00/ton | $1,717,695.00 |
| 15,300 × 0.4039 = 6180 tons<br>6180 tons × $50.80 | $ 314,000.00 |
| Total income—sucrose + sucrose in molasses | $2,031,695.00 |
| *Refining Costs* | |
| 8,379 tons at $11.30/ton | $ 94,500.00 |

**Table 6.—Cost and Income for an Assalini "B" Installation with a Capacity of 30,000 Tons of Molasses per Year.**

| | Cost | Income |
|---|---|---|
| 30,000 tons molasses<br>51% sucrose in molasses (15,300 tons) at $50.80/ton | $ 778,000.00 | |
| *Cost for processing with resins* | | |
| 1,680,000 of 100% NaOH at 4.1 cents/lb.<br>(0.05 lb./lb. of sucrose in molasses or 1.96 lb. molasses) | 69,100.00 | |
| 3,400,000 of $H_2SO_4$ at 1.24 cents/lb.<br>(0.099 lb./lb. sucrose in molasses or of 1.96 lb. molasses) | 42,300.00 | |
| Labor—0.02 cents/lb. of sucrose in molasses | 6,120.00 | |
| *Operating cost—other than resins* | | |
| Labor—0.0785 cents/lb. of sucrose in molasses | 24,050.00 | |
| Fuel Oil (three-effect evaporation)<br>0.274 lb./lb. of sucrose in molasses<br>8,400,000 lbs. of naphtha at 1.09 cents/lb. | 91,500.00 | |
| *Cost of refining* | 94,500.00 | |
| *Interest on resin plant*<br>(10% per year on $96,500) | 9,650.00 | |
| *Amortization of resin plant of 20% per year* | 19,300.00 | |
| *Value of the refined sugar in bags* | | $1,717,695.00 |
| *Value of sucrose in by-product molasses* | | 314,000.00 |
| TOTAL COST | $1,134,520.00 | |
| TOTAL INCOME | | $2,031,695.00 |
| NET INCOME | $ 897,175.00 | |
| | $2,031,695.00 | $2,031,695.00 |

Figure 4 shows a schematic diagram of an industrial plant.

The economic balance is developed in Tables 5 and 6. Here the cost figures refer to Italian practice. The results indicate the advantages to be obtained with the "B" Process in Italy. The extractability of sugar has been based on the equation:

$$\% \text{ theoretically recovered} = \frac{100}{X} \cdot \frac{(X - 0.6)}{(1.0 - 0.6)}$$

where X is the purity of the treated sugar effluent. The value 0.6 stems from the purity of molasses which is the end by-product of the sugar refining operation. No indication of the number of crystallization steps can be given. The figure 0.7867 refers to the purity of the effluent liquor. The value 0.546 was obtained from the above equation using a purity of 78.67%. The figure 0.9980 refers to the moisture content of commecial sugar in Italy. In Italy the weight of the sugar is sold as gross weight in bags so that the factor 0.9912 is used to correct for this value. Commercial value for sugar and molasses refer to average sugar prices in Italy at the time the paper was written.

The cost of refining sugar from the "B" Process liquor is different ($11.30/ton) than for the cost stated in Part I for an "A" Process liquor ($14.20/ton). This is due to the fact that the "B" liquor does not require the coagulation and filtration steps required in the conventional process.

Since this paper was presented, additional work has been reported on these processes. These papers and patents are listed below (1-4). An accurate life estimate for the resin has not been obtained as yet. Pilot plant work indicates no definite trend in operating characteristics for the resin but the amount of cycles still is not sufficient to project the thousands to be expected under life operation.

1. Schiwek, H. 1959. Zeitsch. Zuckerind. 84: 502-508.
2. Schiwek, H. 1960. Zeitsch. Zuckerind. 85: 342-349.
3. Assalini, G. U. S. Patent 2,929,745: Mar. 22, 1960.
4. Assalini, G. U. S. Patent 2,929,746: Mar. 22, 1960.