The Direct Production of Liquid Sugar From Ion Exchange Treated Thick Juice

L. T. Zanto and S. E. Bichsel[1]

Introduction

The recent development by the Holly Sugar Corporation of a secondary ion exchange system which is designed to operate in conjunction with the Hamilton City-type primary ion exchange system (1) will make it possible to produce high quality liquid sucrose on a year around basis from 30 RDS diluted thick juice. Thick juice storage tanks (8), stainless steel evaporators, pressure filters, heat exchangers, and liquid sucrose storage tanks are the other essential elements of the process.

Background and technology of the deionization process as relating to the beet sugar industry in the United States was described by Maudru (5,7) in 1951 publications and updated by Bichsel and Hoover in a 1969 publication (1).

Since the early days of the deionization process, many of the pitfall conditions, including poor equipment design and inferior resins, have been improved. Recent developments in by-product recovery and liquid sugar production as well as declining beet quality should necessitate the use of more ion exchange purification in the future.

Production of liquid sucrose on a year around basis from thick juice by deionization is particularly inviting at factories which are close to metropolitan areas and distribute a substantial portion of their production in the liquid form to canners, bottlers, and dairies. Other probable advantages for this process are:

1) Increase of factory extraction through nonsugar elimination.
2) Requirement of less sugar-end equipment.
3) Storage of sugar as thick juice and liquid sugar rather than in more expensive silos and warehouses.
4) Utilization of the by-product RNS (reconstituted nonsugars) as a feed supplement for beet pulp and the salty acidic ammonium sulfate waste regenerant stream as a valuable fertilizer by-product.
5) Realization of a faster pay-off of resin and equipment by continuous operation.

Discussion

Fundamental to the production of liquid sucrose from a process stream is a working knowledge of marketing requirements.

[1] Research Chemist and Driector of Research, respectively, Holly Sugar Corporation, Colorado Springs, Colorado.

[2] Numbers in parentheses refer to literature cited.

From this knowledge, evaluation of a number of processes and process streams may be made. Based on the research in this area, conclusions affecting the development of a process are:

1. The previously successful utility of the Hamilton City-type deionization system could provide an adaptable foundation for further development.
2. It is difficult to achieve a salable high quality product using highly colored and law purity feeds such as diluted molasses or machine syrup in a two-stage deionization system.
3. The weak ionic nature of removable nonsugars in Hamilton City effluent streams suggests the use of strong acid and strong base secondary exchangers (6).
4. Cane sugar processors have found it possible to make liquid sucrose from raw sugar by the deionization process (2,3,4).
5. With attention to process control in utilizing the Hamilton City-type deionization system, a 98 apparent purity effluent can be acquired from dilute thick juice of at least 88 apparent purity and no more than 600 color units (ICUMSA method - 560 mμ).
6. Further purification of the effluent from the Hamilton City system by means suggested in Conclusion 3 is necessary to reduce ash and color values.
7. A high quality effluent has been successfully produced on an experimental scale by using primary and secondary separated monobed columns containing strong base, type II, anion exchange resin overlaying strong acid cation exchange resin.
8. Evaporation of secondary deionization effluent under conditions of pH 7.0 - 7.5, temperaure - 140° F, 20 inches Hg vacuum, resulted in a high quality product.

In Figure 1, a schematic for liquid sucrose production is shown. The steps in sequence are thick juice storage, primary deionization system, secondary deionization system, and evaporation. Thick juice storage, filtration, cooling, and product storage have been successfully utilized by the Holly Sugar Corporation as well as other companies in this country and Europe.

The Hamilton City-type primary system is comprised of three columns containing a strong acid cation exchange resin and three columns containing a weak base anion exchange. The cation exchange resin is regenerated with 6 to 10% sulfuric acid while the anion exchange resin is regenerated with 4 to 6% aqua ammonia.

For the production of liquid sucrose, 87 to 88 apparent purity - 30 R.D.S. diluted thick juice is cooled to 8°C and passed at a flow rate of .30 gallons/minute/cu. ft. of cation exchange resins, through a cation exchange column which is in series
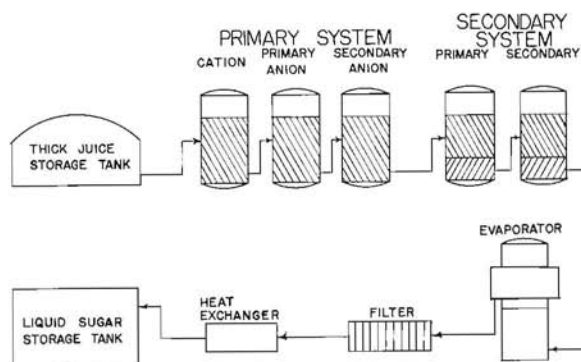
Figure 1.—Flow diagram for the direct production of liquid sugar by deionization.

with a primary and secondary anion exchange columns. The other cation and anion exchange columns are either in the regeneration or stand-by phases. The service cycle duration of the cation exchange columns is determined by the effluent conductivity while the anion exchange columns' service cycles are determined by the pH of the secondary effluent.

While the development of a secondary deionization system involved the screening of several resins and process approaches, a high quality effluent was produced by a merry-go-round system (refer to discussion of Hamilton City-type anion exchangers) of three separated monobed columns (jacketed columns of 2-inch inside diameter by 48-inch length) which contained two parts of strong base, type II, anion exchange resin overlaying 1 part of strong acid cation exchange resin.[3]

Effluent of 22 R.D.S. and 12°C from the Hamilton City system was passed downflow through primary and secondary columns connected in series at a flow rate of .33 gallons per minute per cu. ft. of resin with respect to the primary column. Effluent flowed through a conductivity cell and was recorded by a Beckman Electronik 15 recorder. Samples were adjusted from pH 3.5 - 4.5 to neutral for analyses and evaporation.

Effluent conductivity ranged from 5 to 20 microhms/centimeter for several bed volumes but a regenerated secondary column was placed on stream when the conductivity reached 60 microhms/centimeter.

In Figure 2, solution grade color and percent conductivity ash in the effluent are plotted versus bed volumes of throughput with respect to one column. For the first 13 bed volumes, only a primary secondary-system column was on stream. Regenerated

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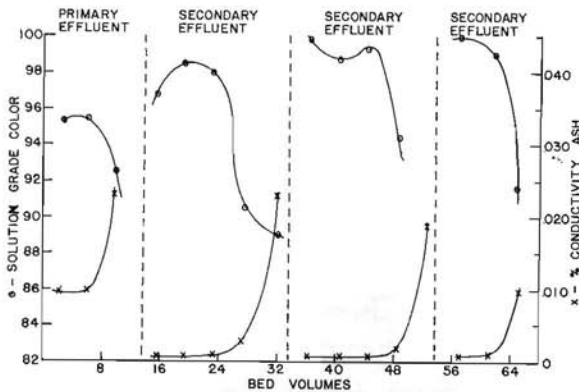[3] Information on resin tests may be obtained from the authors.

Figure 2.—Graphic relationship of solution grade color and % conductivity ash to bed volumes of concentrated secondary deionizer effluent.

secondary columns were placed in service after 13, 34, and 53 bed volumes of effluent throughput. It is of particular interest to note the sharp conductivity ash and color breakthrough.

The anion resin was regenerated with 4 percent sodium hydroxide and the cation resin with 10% sulfuric acid. Success in retaining capacity was realized by stripping color and impurities from the bed prior to regeneration with a dilute sodium chloride solution.

The choice of a separated monobed column which operates in merry-go-round was based on the following information.

1. While many of the remaining impurities are amphoteric, they are more easily removed by cation resin. By removing the impurities with a cation resin, the effluent becomes quite acidic and subsequent passing of cationic effluent through a strong base anion resin does not neutralize the juice. Addition of base to neutralize the solution results in a high conductivity ash.

2. Mixed beds show the same results as those of point 1.

3. By passing feed juice through the strong base anion exchange resin and then the strong acid cation exchange resin, the anion exchanger is forced to remove as many amphoteric impurities and color bodies as possible and then the cation exchanger removes the remaining impurities. Effluent pH's range from 3.5 to 4.5 but the solution is unbuffered and requires a very small amount of base for neutralization.

4. A 2:1 ratio of anion exchange resin to cation exchange resin provides the best exhaustion balance.

5 Odorous amine groups which are lost by the anion resin are removed by the cation resin.

6. A more complete bed exhaustion and higher quality product are realized by operating in merry-go-round.

Concerning evaporation, it is important that color formation is minimized. Effluent from the secondary system, pH 3.5 - 4.5, because of invert formation must be adjusted to neutral or to some point in the low alkaline range. It is also known that color is formed from the degradation of invert in the alkaline range at temperature sufficient for evaporation.

In order to determine the color formation at pH values in the low alkaline range, solutions containing 65 percent sucrose, .50% glucose, and .50% fructose were prepared in buffered water (pH 7.0 - 7.5 - 8.0 - 8.5 - 9.0) and heated to 80° C on an agitated water bath. Color formation versus time is shown for the 5 solutions in Figure 3. This test indicates that the effluent should be adjusted to pH 7.0 - 7.5 before evaporation.

Because of the substantial color formation in high pressure, long tubed evaporators, it is necessary to design a system which operates at low temperature and pressure.
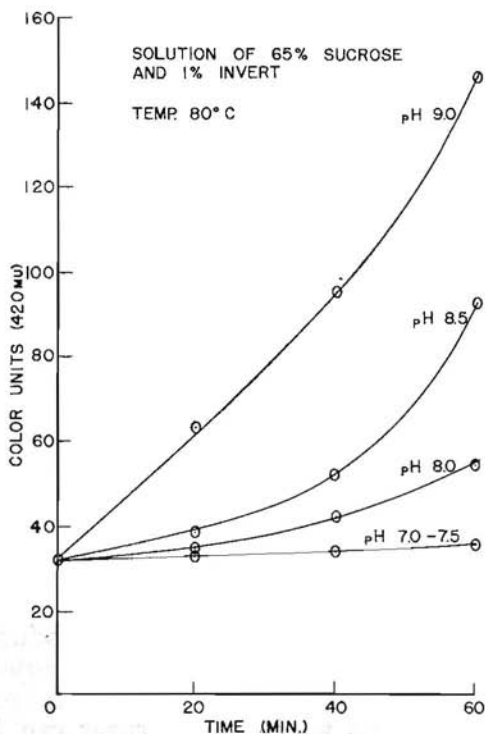

Figure 3.—Relationship of color formation to time for 65% sucrose - 1% invert solution with pH values in the low alkaline range when heated to 80° C.

Summary

Results obtained from processing 30 R.D.S. diluted thick juice with the Hamilton City deionization system and the previously described secondary system are shown in Table 1. Effluent from the secondary system was concentrated (temperature - 140° F., 20 inches Hg vacuum, to 67 R. D. S.).

Table 1.—Comparative analyses of thick juice, primary deionized effluent, and concentrated secondary deionized effluent.

	Thick juice feed	Primary effluent	Conc. secondary effluent
A.P.	91.0	98.0	98.1
Invert	.70	1.31	1.40
Raffinose	.40	.40	.40
Total N	.70	.07	----
Na	.45	.15	----
K	1.10	.003	----
Cl	.35	.11	----
Color/turb.	600*	295**	94-100***
pH	8.9	8.5	7.0
Cond. Ash	. --	----	.007

* ICUMSA 560 mu
** ICUMSA 420 mu
*** Holly Solution Grade

The apparent purity of the beet and primary effluent are analyzed values while that of the concentrated effluent is 100 minus the contribution of invert and raffinose.

Invert in the final product can be reduced from 1.46% to .70% on sugar by more complete invert destruction in carbonation and operation of the primary system more effectively. Raffinose remains constant through the process.

Total nitrogen, sodium, potassium, and chloride are eliminated as is indicated by the low conductivity ash in the product. The conductivity ash of .007% is well below normal required values.

The product solution grade (color-turbidity) of 94 to 100 is considered excellent. Complete elimination of floc (saponin) is accomplished with ion exchange treatment.

From Table 1, it is evident that the direct production of liquid sucrose from thick juice by deionization is feasible. Before an economic analysis which compares the direct production by the deionization process with melting sugar can be presented, additional studies on strong base anion resin including chemical - physical stability, fouling, and regenerant usage must be completed.

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