Ion Exchange Operation at Two Beet Sugar Factories in Japan

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Background Information

In Japan there are ten beet sugar factories, most of which are located in Hokkaido. Hokkaido is the second largest of the Japanese islands. It is located in the north (about 42° to 46° north latitude and 140° to 146° east longitude). Aomori Prefecture, the beet-growing area of Honshu, faces the southernmost section of Hokkaido.

The quantity of sugar produced in these beet sugar factories totaled about 173,000 tons during 1964-65 campaign. This represented only 11% of the total sugar output in Japan. Thus the beet sugar industry occupies a relatively minor position in Japan. This is because beet cultivation came to be encouraged by the government after the war as a means of promoting agriculture in the cold regions of northern Japan; the industry is therefore not yet widespread.

Ion exchange is used extensively in the cane sugar refining industry in Japan. Nearly all of the sugar refineries use chloride-cycle anion exchange to decolorize fine liquor. In beet factories also, ion-exchange resin is being used to decolorize and deionize sugar juice.

In Japan, the ratio of the price of molasses to refined sugar is low as compared with that in the United States and Western Countries. This is because other raw materials for fermentation industries (potato-starch, imported dried jujube, etc.) can be obtained cheaply. Therefore, the beet factories in Japan have a particularly high incentive to recover as much sucrose from the beets as possible. For this reason, all the existing beet factories had been using the Steffen process.

In recent years, studies have been made on demineralization by ion-exchange resins as a means for increasing sugar yield. As a result of these studies in Japan, the economic prospects were highly encouraging and five plants were installed. Thus, half of the beet sugar factories there are now using ion-exchange.

Ebara-Infilco Company Ltd., as a result of its study of ion-exchange demineralization processes best suited to the present

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situation in Japan, conceived a process which combines conventional deionization of cooled second carbonation juice with partial decolorization. After conducting pilot plant tests during one campaign, a full-scale installation incorporating this method was made at the Fuji Seito (Sugar Refining) Company Ltd., Aomori Factory, in 1962. A similar process was installed at the Dai-Nippon Sugar Manufacturing Company Ltd., Hombetsu Factory, in 1963. The nominal beet slicing capacities at these two factories are 1400 and 1500 tons per day, respectively.

Fuji Seito Company, Aomori Factory

Preliminary remarks

This factory started operation in 1962. In the planning of its construction, the company conducted comparative studies on the conventional Steffen process and ion-exchange demineralization process in order to decide which to adopt. As a result of such study, it was concluded that the ion-exchange method is more suitable for the following reasons:

A. Beets grown in and around Aomori Prefecture (where the factory is located) have relatively low sucrose and high raffinose; thus the yield by the Steffen process would be limited.

B. At the beginning of the campaign, the temperature of cooling water would be relatively high, requiring refrigeration for the Steffen process. (Cooling is more critical for the Steffen process than for ion-exchange.)

As for the choice of ion-exchange method, comparative studies were made on the Imacti, BMA and Quentin processes. The company finally decided to adopt the conventional cold deionization system with decolorization partially added (developed by Ebara-Infilco Company).

During the 1962/63 campaign, the factory worked with a system of three cation exchangers and two anion exchangers. This experience indicated the need for a third anion column for more efficient operation. This was installed for the second campaign (1963/64). Furthermore, another cation column was erected after the second campaign to recover betaine from the waste regenerant of the cation exchanger. By now, three campaigns have gone by since the factory commenced operation. As anticipated, the beet pol was low (14.4%, 15.5%, and 14.55% in respective campaigns); however, the objectives of the ion-exchange station with respect to product quality and yield was achieved, indicating that the ion-exchange equipment was highly effective.

Flow diagram

The general process flow diagram for beet sugar production at Fuji is shown in Figure 1, and that for the ion-exchange process is shown in Figure 2. Sulfur dioxide is added to the second carbonation juice which is then filtered through a ceramic filter. This thin juice which is fed into the ion-exchange system is referred to as "original juice." The original juice is bifurcated into two lines, one to be deionized after cooling and the other to be decolorzed without cooling. With respect to the volume ratio of each line to original juice, one must consider the quality of original juice to be treated and the purity of thick juice. The average percentage of decolorized juice against total original juice in each campaign was 25.2%, 32.2%, and 17.0%, respectively, from 1962/63 to 64/65.

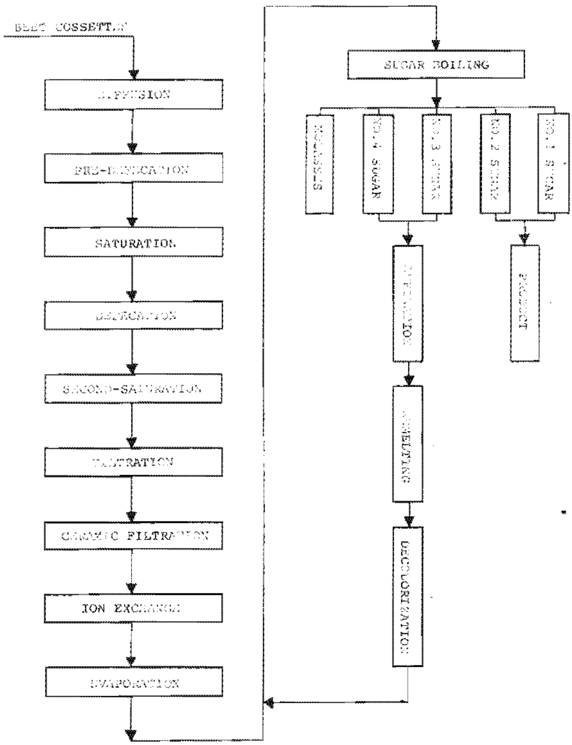

Figure 1.—General flow diagram of beet sugar production at Fuji Seito Company.

In operating the decolorizing units, a portion of the hot thin juice is fed into the decolorizing units which contain strong-base resin in the chloride form. It should also be mentioned that

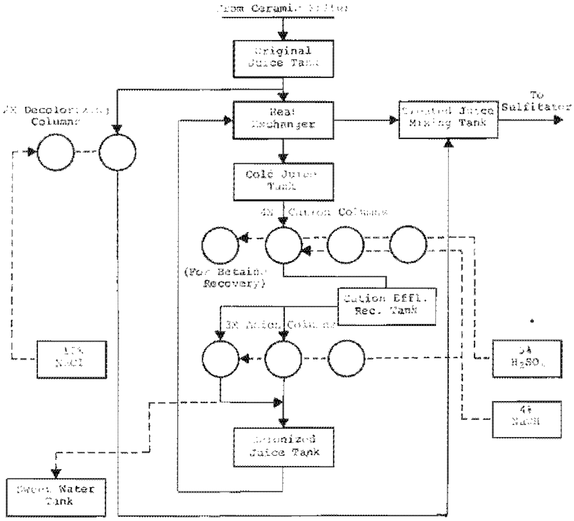

Figure 2.—Flow diagram of ion exchange process at Fuji Seito Company, Aomori Factory.

remelt sugar is also decolorized by chloride-cycle anion exchange. The resin for this application had been previously used for decolorizing in a cane refinery.

The other portion of original juice flows through a plate heat exchanger (made by De Laval) to maintain juice temperature below 13°C, then into the cation exchanger. The volume of cation effluent is divided into two equal portions, both of which are fed to two anion exchangers in parallel. The anion exchange units contain epoxy-polyamine intermediate-base anion exchange resin regenerated to the free-base (OH) form. The effluent from the anion exchanger (approximately 280 gpm) flows again into the heat exchanger to raise its temperature and is mixed with decolorized juice. The combined effluent liquor is referred to as "mixed juice." This mixture is treated with SO_2

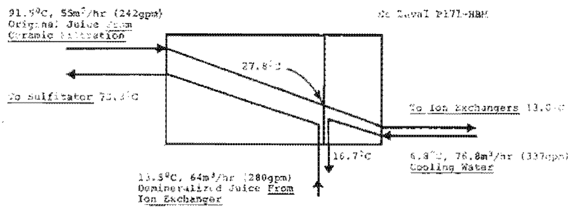

Figure 3.—DeLaval heat exchanger operating data.

and is pH-controlled in order to prevent subsequent decomposition and recolorization. It is then fed into quadruple-effect evaporators.

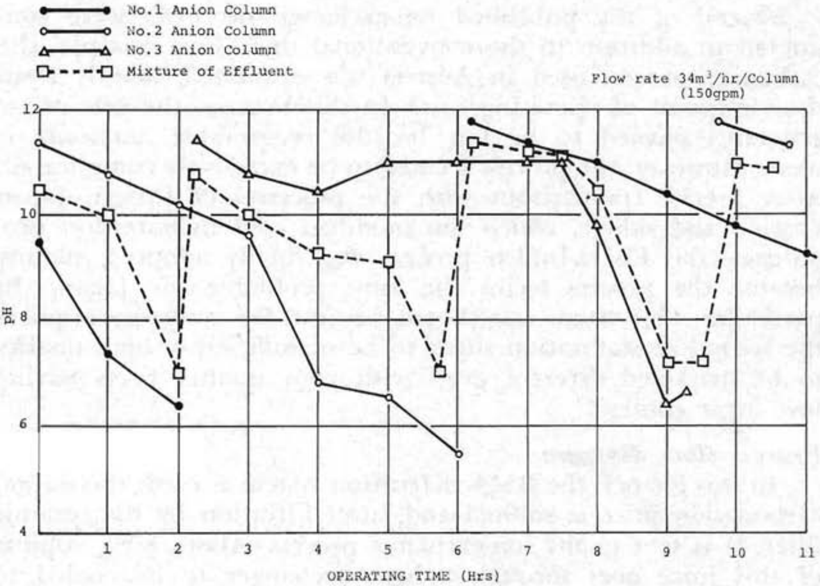

Figure 4.—pH curve of anion effluent in parallel operation (Fuji).

Dai-Nippon Sugar Refining Company, Hombetsu Factory

Preliminary remarks

The Hombetsu factory was built in 1962. It started without any sugar recovery process, but ion-exchange was contemplated for the start of the second campaign, 1963/64. Precise economic comparisons of ion-exchange with the conventional Steffen process were made, and the former was decided to be superior for the following reasons:

A. By the use of ion-exchange, the indicated sugar recovery was about the same or slightly greater than with the Steffen process.

B. The lower the quality of beets, the more effective and economical the ion-exchange process.

C. The operational and housekeeping facilities of the Steffen process were deemed to be more troublesome and less efficient from the sanitary point of view; it was considered more difficult to treat the waste and lime cake than the ion-exchange regenerants.

D. If the Steffen process were adopted, it would be necessary to add decolorizing equipment for the thick juice in order to obtain the required high quality in the crystalline sugar product.

Several of the published ion-exchange processes were considered in addition to the conventional one. For example, the carbonate process used in Austria was examined, mainly from the viewpoint of operating cost. In this process, the cost of regenerants seemed to be low because recoverable ammonia is used. However, the process seemed to be excessively complicated. After precise comparisons with the processes of Imacti, Japan Organo and others, which use modified cold deionization procedures, the Ebara-Infilco process was finally adopted, mainly because the process seems the most profitable for Japan. In particular, the system was chosen because the company requires the second crystallization sugar to be of sufficiently high quality to be marketed directly, even with poor quality beets having low sugar content.

Process flow diagram

In this factory, the BMA defecation system is used; the second carbonation juice is sulfited and, after filtration by the ceramic filter, it is sent to the ion-exchange process. About 80% volume of this juice goes through a heat exchanger to be cooled to about 11°C and fed into successive columns of cation/anion/anion

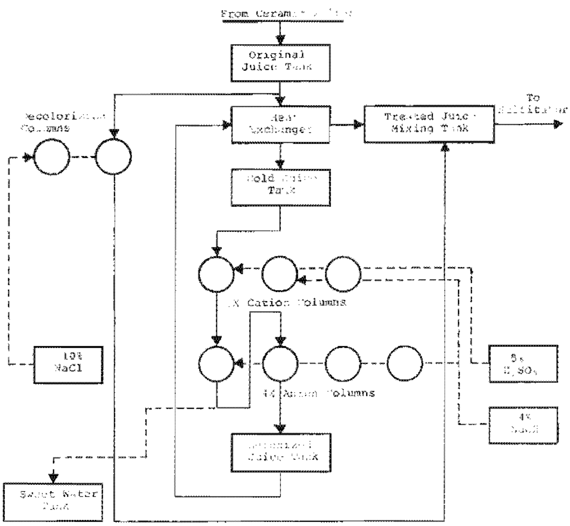

Figure 5.—Flow diagram of ion exchange process at DaiNippon Sugar Manufacturing Company, Hombetsu.

resins. The remaining 20% portion of hot original juice is decolorized by the use of strong-base anion-exchange resin in the chloride cycle. The deionized juice recovers heat to 75°C through the heat exchanger and it is then mixed with the decolorized juice. Sulfur dioxide is added to stabilize the juice prior to evaporation (Figure 5).

Results and Discussion

Resin volumes and regenerant dosages at both factories are indicated in Table 1. It should be noted that small quantities of caustic soda are used regularly to cleanse the cation exchangers prior to regeneration with acid.

Operating data and product quality at Fuji are shown in Tables 2a, 2b, and 2c. The deionization process removes more than 90% of the ash and an average of 86% of the color. Although the decolorizing units removed less color than the deionizer, the blending of the two streams provided a desirable buffering effect—which prevented subsequent adverse effects in the evaporators.

The high invert content of the mixed juice during the 62/63 campaign was due to bacterial fouling of the heat exchanger and to channeling in the anion exchange columns. These difficulties were corrected prior to the 63/64 campaign. Channeling was eliminated by a very inexpensive and simple bentonite treatment.

In considering the average quality of products (Table 2c), one should bear in mind that these were obtained with first and second strike sugars, both of which were converted directly to product.

The data from the Dai-Nippon (Hombetsu) factory are given in Tables 3a, 3b, 3c, 3d, 3e, 4 and 5. These data are particularly interesting inasmuch as they allow comparison of results without (62/63) and with (63/64 and 64/65) ion-exchange. The expected lowering of ash content and color improvement were obtained by ion-exchange. However, the very high invert content of the thick juice during the 62/63 campaign (Table 3a) was due to adverse conditions during the first year of operation and had nothing to do with the absence of ion-exchange.

Tables 3a and 3b show the relatively small increase in invert by the deionization process. This reflects the effective cooling of the thin juice.

The nitrogen removal data (Table 3d) are particularly noteworthy in that they reflect the efficiency and reversibility of the porous cation exchanger, Duolite C-25, in adsorbing ammonia.

Table 1.—Process specifications.

	Resins used (No. columns)			Resin volume in each column, cu ft			Regeneration dosage, lbs/cu ft			
							Cation		Anion	Decolor.
Factory	Cation	Anion	Decolor.	Cation	Anion	Decolor.	100% H_2SO_4	100% NaOH	100% NaOH	100% NaCl
Fuji	Duolite C-25 (4)	Duolite A-30B (3)	Strong-base (2)	335	440	45	6.25	1.25	3.9	9.4
Dai-Nippon	Duolite C-25 (3)	Duolite A-30B (4)	Strong-base (2)	317	264	65	6.25	1.25	3.9	9.4

Table 2a.—Average operational data—ion exchange process (Fuji Seito Co.).

	Original (thin) juice					Deionized juice (anion eff.)						Mixed juice					Decolor. juice
Campaign	BX	App. pur.	Invert, % on Bx	% Ash on Bx	Color °St	BX	App. pur.	Invert, % on Bx	Tr. pur.	% Ash on Bx	Color °St	BX	App. pur.	Tr. pur.	Invert, % on Bx	Color °St	Color °St.
62/63	11.8	91.7	0.17	1726[1]	15.2	10.1	98.6	0.30		137[1]	2.5	10.6	97.4		0.47	4.1	4.8
63/64	12.0	90.6	......	0.296	12.1	10.2	98.7		97.6	0.016	1.2	11.2	95.4	94.4	0.26	4.1	7.4
64/65	11.1	89.9	0.09	2029[1]	11.4	9.6	98.4	0.31	96.8	197[1]	1.7	10.0	96.6	95.0	0.33	2.1	3.3

[1] Micromhos/cm

Table 2b.—Average operational data in the evaporators.

Campaign	Evaporator juice temp. °C I	II	III	IV	Thick juice quality App. pur.	Invert % on Bx	Color °St
63/64	116	107	99	77			—
64/65	119	112	107	81	96.6	0.19	2.5

Table 2c.—Average quality of products.

	Moisture %	Pol %	Invert, % on Bx	Color °St[1]
62/63	0.027	99.9	0.02	—
63/64	0.034	99.9	0.02	0.26
64/65	0.040	99.9	0.02	0.21

[1] With 50° Brix solution

Table 3a.—Average operational data without and with ion exchange (Dai-Nippon).

Campaign	App. purity Original	App. purity Thick	Invert sugar % on Bx Original	Invert sugar % on Bx Thick	Ash % on Bx Original	Ash % on Bx Thick	Color value °St Original	Color value °St Thick
62/63[1]	91.7	91.8	0.14	0.74	—	2.06	15.3	22.4
63/64	93.3	97.8	0.09	0.23	1.83	0.41	13.5	1.58
64/65	93.4	97.2	0.14	0.27	1.81	0.51	15.0	2.3

[1] No ion exchange

amino compounds and betaine. As expected, the pyrrolidone carboxylic acid (PCA) is removed by the anion rather than the cation exchanger. The total nitrogen leakage shown in the anion effluent (0.051% on Brix) is probably due to colloidally dispersed protein—which escapes adsorption.

With respect to product quality (Table 3e), the principal advantage shown by the deionized product was in the solution color; the treated sugar develops less than half as much color as that which did not have the benefit of ion-exchange. Again, it should be noted that the product comes from the first and second strikes combined.

Perhaps the most significant result obtained (Table 4), was the increased product yield and decreased molasses yield, particularly in view of the very low molasses prices existing in Japan. More precise calculations of extraction must await the development of more accurate methods for determining the net sucrose content of the beets[2].

Although a complete cost analysis is not yet available, Table 5 shows the regenerant consumption and the estimated costs of these regenerants to produce 100 pounds of sugar. Since the

[2] For more information on sugar extraction, the reader is invited to contact the authors or the Dai-Nippon Sugar Manufacturing Company, Engineering Section.

Table 3b.—Monthly average operational data (1964).

	Original juice				Deionized juice			Decolorized juice		Mixed juice			
	Bx	App. pur.	Invert % on Bx	Color	Bx	App. pur.	Invert % on Bx	Bx	Color	Bx	App. pur.	Invert % on Bx	Color
Oct.	13.5	93.1	0.21	17.6	11.9	98.1	0.26	13.3	4.1	12.2	96.4	0.24	1.9
Nov.	10.2	93.1	0.14	14.3	12.3	97.9	0.27	14.1	4.7	12.5	97.0	0.22	2.7
Dec.	13.5	93.7	0.11	14.9	12.1	98.9	0.16	13.3	6.7	11.7	97.7	0.16	3.3
Avg.	13.7	93.4	0.14	15.0	12.2	98.4	0.22	13.6	5.5	12.1	97.0	0.20	2.8

Table 3c.—Average operational data in the evaporators (juice temp. °C).

	1	2	3	4
62/63	129	120	109	95
63/64	114	109	104	83
64/65	120	118	105	76

Table 3d.—Nitrogen removal by ion exchange.

	Total—N % on Bx	Ammonium—N % on Bx	PCA—N, % on Bx	Amino—N % on Bx	Betaine—N % on Bx	Betaine, % on Bx
Original Juice	0.489	0.052	0.060	0.087	0.125	1.047
Cation Effluent	0.131	0.000	0.056	0.006	0.000	0.000
Anion Effluent	0.051	0.001	0.001	0.004	0.000	0.000

Table 3e.—Average quality of products.

Campaign	% Moist.	Pol.	% Invert	% Ash	Color Value: Solution[1]	Color Value: Reflectance[2]	Grain size (mm): Mean aperture	Grain size (mm): Coeff. of variance
62/63	0.04	99.8	0.02	0.02	47.0	79.3	0.45	0.15
63/64	0.05	99.9	0.01	0.01	21.2	82.4	0.47	0.12
64/65	0.05	99.9	0.02	0.03	25.4	82.5	0.48	0.12

[1] International Commission for Uniform Methods of Sugar Analysis.
[2] Reflectance using blue filter.

Table 4.—Data of sugar yield (Dai-Nippon).

Campaign	Beet pol.	Product yield	Molasses yield
62/63	16.15	12.55	4.41
63/64	16.75	14.55	1.37
64/65	16.83	14.87	1.39

Table 5.—Regenerant consumption and cost.

Campaign	Lbs regenerant per cwt. sugar produced: NaOH 100%	Lbs regenerant per cwt. sugar produced: H_2SO_4 100%	Lbs regenerant per cwt. sugar produced: NaCl	Regen. Cost[1] per cwt. sugar prod.
63/64	3.05	4.49	0.21	$0.211
64/65	3.27	4.25	0.29	$0.218

[1] Based on Japanese prices.

regenerant costs are about 60% of the total ion-exchange costs in treating cooled thin juice, it is reasonable to assume that the overall fixed charges against ion-exchange are less than three dollars per hundredweight of bonus sugar in the bag. It should also be mentioned that these costs are based on Japan prices and that caustic soda is about 25% higher than in the U.S.

No doubt these costs could be lowered significantly by recovering a portion of the acid for re-use and by using ammonia for regeneration instead of caustic soda. Ammonia regeneration is particularly attractive because of its fertilizer value after use and/or the ease of recovering it for re-use by reaction with the cheaper lime.

Summary

In both factories, the Fuji Seito Company, and the Dai-Nippon Sugar Manufacturing Company, the deionization of beet sugar thin juice by the use of Duolite C-25 and Duolite A-30B in series has resulted in high quality sugar production and good yields, as expected.

Duolite C-25, a porous cation exchange resin, adsorbed a major portion of the nitrogenous compounds and some of the color bodies contained in the "original juice". These materials were completely eluted by the use of caustic soda solution followed by acid regeneration. No deterioration of cation resin performance was observed. A betaine recovery process was developed by Fuji Seito by the use of Duolite C-25 in combination with modified regeneration.

The anion exchanger, Duolite A-30B, performed well in removing mineral acids and color. Pyrrolidone carboxylic acid was also effectively adsorbed by this resin.

As expected, the salt-splitting capacity of the anion exchange resin declined as the resin aged in service. As a result, the pH of the anion effluent dropped at the end of each cycle; thus the volume of treated juice (per cycle) to pH 7 decreased from the start to the end of each campaign. In order to keep the pH-value of the anion exchange effluent above 7 at the Fuji plant, the cation exchange effluent was split and fed into two anion exchangers in parallel. At the Dai-Nippon plant, two anion exchangers were operated in series, and as a consequence, the pH of the anion exchanger effluent was kept above 7 throughout the campaign. Also the nonsugar removal capacity per column was increased in this way.

A few problems remain to be solved. For example, some attrition loss was encountered with the anion-exchange resin. A new and improved epoxy-polyamine resin, Duolite ES-57, gave encouraging results in our small pilot test, and will be tried as a replacement for A-30B.

Acknowledgment

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