De-Ionization and Beet Sugar Production at the Layton Sugar Company

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THE RECENT development of the resinous type ion-exchange materials and the stability of these resins to wide ranges of operating conditions has opened new fields to their application; not the least of these is in the field of sugar production.

After several years of investigational work in our own laboratories on this problem, it was felt that further work must of necessity be done as pilot-plant studies in the field. Accordingly arrangements were made with the Layton Sugar Company to conduct pilot-plant studies in their plant during the 1946 campaign. Results of these studies confirmed the laboratory findings and permitted the drawing of the following conclusions: 1. more high-purity sugar per ton of beets may be delivered to the bag with less reprocessing; 2. inversion can be held to a minimum if temperatures are properly adjusted; 3. the quantity of final molasses will be materially reduced, and what molasses there is will be an edible product; 4. the investment in full-sized equipment appears to be economically sound.

In view of the results obtained from this pilot-plant study the Layton Sugar Company contracted for an installation to treat their entire production of 1,350 tons daily capacity. Installation was to be completed in time for the 1947 campaign. When final arrangements were completed less than 6 months remained to complete the job of designing, fabrication and erection of the entire plant. Little wonder it was not completed when slicing operations started on October 7. The de-ionization plant operation started on October 22.

The de-ionization plant consisted of four units each unit being composed of a cation cell 10 feet by 14 feet containing approximately an 8-foot bed of resin and an anion cell 10 feet by 11 feet containing approximately a 5½-foot bed of resin. A valve assembly was provided on each unit, with adequate headers, to direct the flow of liquids during the various phases of operation. The valves are hand-operated, rubber-lined Saunders-type diaphragm valves. Common to all units is a set of regenerant tanks with automatic controls for refilling and diluting the regenerants. Pumps are provided for pumping the regenerants through the exchanger beds. Controls such as rate-of-flow indicators, conductivity meters, specific-gravity recorders, positive-displacement meters, recording thermometers, etc., were also provided. A duplex water softener was provided to supply soft water for diluting the ammonium hydroxide regenerant and for regenerating the acid-adsorber beds. This prevents the precipitation of magnesium hydroxide on the resin bed which would lengthen the regeneration period. All tanks,

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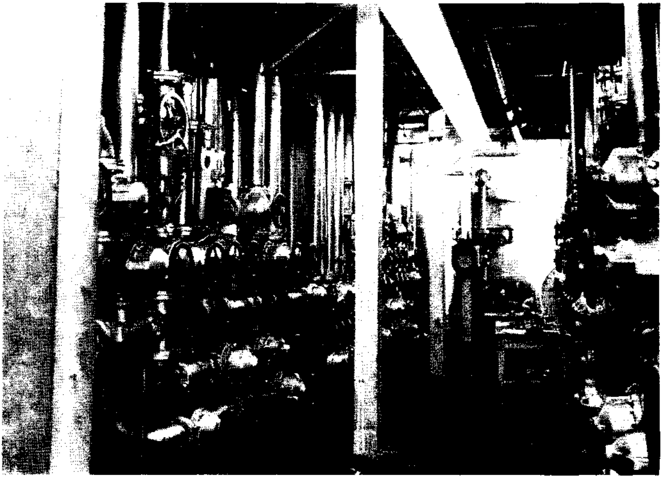
valves, piping, etc., that come in contact with acid, liquor and juices were rubber lined.

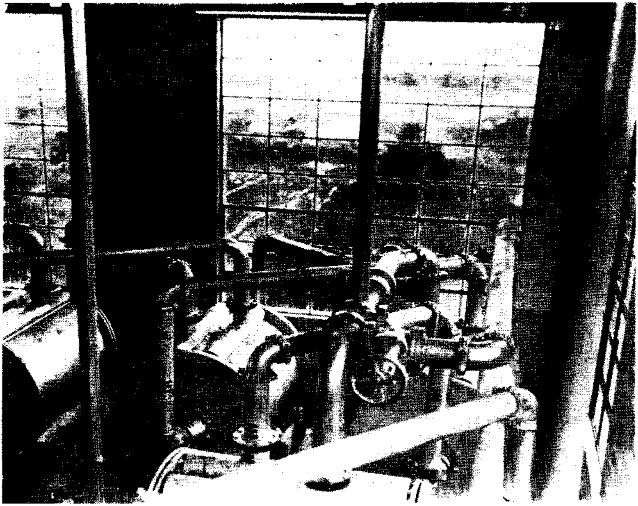
Bulk handling equipment for unloading and storing of carload lots of concentrated acid and ammonium hydroxide were a part of the installation.



Six spiral heat exchangers were installed to cool the incoming juice and reheat the treated juice.

The operation of this equipment was relatively simple. After approximately 1 weeks' training period one man and a helper per shift conveniently operated the station. In fact on occasions, one man operated the station alone.

During pilot-plant studies it was found that excessive inversion was experienced if the juice was passed through two units in series in order to remove the last traces of ionizable salts, therefore, the plant was designed for single pass operation. Each unit was designed to handle the entire plant production for at least 1¾ hours. This gave 5¼ hours from the time any unit was shut down till it was needed for service again. This procedure allowed more than sufficient time to regenerate each unit. Graph 1 shows the sequence of operation of the four units.



Second-carbonation juice was used as the raw supply to the de-ionization station. This juice first passed through the six heat exchangers, where it was cooled, and the treated juice reheated prior to passing on to the evaporators. It was hoped that the juice could be treated at a temperature of 50 degrees Fahrenheit or less, but the supply of cold water was somewhat limited and the temperature occasionally reached 60 degrees to 65 degrees Fahrenheit. There has been considerable discussion as to the possibility of treating raw diffusion juice in order to save the cost of liming and carbonation. There is always the question of removing the colloids from the raw juice and it is felt that possibly the lime is the cheapest filtering medium obtainable. A new approach to the problem of filtration has recently been presented, however, that may change this picture in the near future. More experimental data must be obtained before a definite answer can be given.

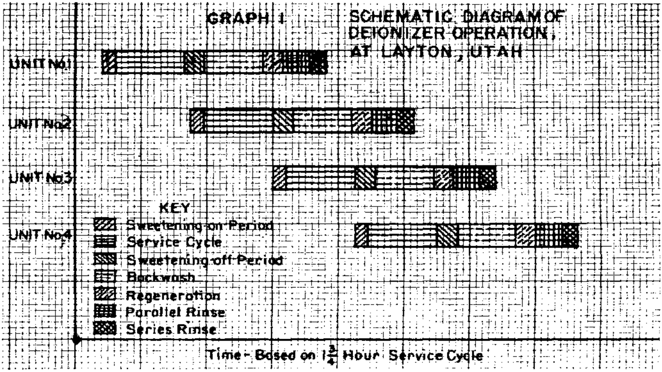
After passing through the ion-exchange process, the juice is practically water white, bland tasting and has completely lost that "beety" odor.

Needless to say, this high-purity juice created some operating problems in the factory, but since another paper is to be presented on this phase of the project, they will not be discussed at this time.

The complete cycle of operation of a unit consists of the following steps:

1. Sweetening-on.
2. Service.
3. Sweetening-off.
4. Regeneration.

At the start of the cycle the voids in the resin beds are full of water. As juice is started to the unit some 70 to 80 percent of this water is forced from the bed containing no sugar and may be discarded or returned to water reclaim. The remaining voids water becomes increasingly stronger in sugar content until the water is displaced and the juice passes through the unit at full sugar strength. This step is known as sweetening-on.



Normally the flow of juice is continued through the unit until there is evidence of the resins becoming exhausted. In this instance, an attempt was made to anticipate this break and start the sweetening-off step before the resins were exhausted so that all the juice in the unit could be forced ahead rather than recycled through a fresh unit. This procedure worked out very satisfactorily.

After the specified amount of juice had passed through the unit, a new unit was turned into service and water turned into the exhausted unit forcing out the remaining juice. Approximately 80 to 85 percent of this juice is forced out at full sugar content. The remaining juice is forced out at decreasing sugar content. When the sweet water is of such low brix that it is no longer practical to evaporate the water for the small amount of sugar remaining, the water is turned to the sewer.

After sweetening-off the cation and anion resins are backwashed individually. This serves the dual purpose of first removing any colloidal matter or sediment that has collected in the beds during the service cycle and second to loosen and reclassify the beds. It has been found that definite advantages are to be had if this backwash is done with hot water. The hot water will more easily loosen and remove certain waxy and gelatinous materials found in the beds. By proper selection of resins this can be accomplished without detrimental effects on the life of the resins.

The resins are now ready for regeneration. The cation unit is regenerated with a 4 to 5-percent solution of sulfuric acid and the anion unit with a 5-percent solution of ammonium hydroxide. Each regeneration required 1,600 pounds of 66 degrees Be' acid, 1,095 pounds of anhydrous ammonia and approximately 100,000 gallons of water of which some 15-20,000 gallons may be recycled for a net water consumption of 80-85,000 gallons. Approximately 30 percent of the acid is reclaimed and used as the first acid on the next regeneration. The remaining acid regenerant and the ammonia solution is discharged to the sewer.

It is believed that the quantities of regenerants used were somewhat excessive and that a substantial reduction can be accomplished without impairing the efficiency of the operation. The urgency of other work prevented a detailed study of this point during the past campaign but it is hoped that a reduction of some 25 to 30 percent can be accomplished this next year.

Considerable work has been done in our laboratory on the problem of recovering valuable by-products from the spent regenerant solutions but more work must be done before a definite statement can be made. It is entirely within the scope of imagination, however, that the major part of the operating expenses may some day be carried by these by-products.

After the regenerants are introduced the beds are rinsed, first individually and finally in series, to remove the last traces of excess regenerants. The unit is now ready for another operating cycle.

From October 22 when the ion-exchange equipment went into service, it was in continuous operation to the end of the campaign on December 25, with the exception of approximately 36 hours of which the greater part was due to failure of the supply of ammonia and a relatively short shut-down for cleaning heat exchanger equipment. After approximately a month's operation a decrease was noted in the anion capacity. We were aware that the other plants had and were encountering the same difficulty but nonetheless we were disturbed. It should be pointed out that the difficulties were with a relatively low pH effluent but not with a low overall capacity of the resin for strong acids. Stopgap measures of correction were introduced but it was of the utmost importance to uncover proper permanent correctional procedures.

Such experiments were made as could be performed in the Sugar Company's laboratory to restore this capacity but they were only partially successful. It was, however, well demonstrated that the loss was not due to any destruction of the resin but rather to the cumulative effect of trace organic compounds in the beet juice irreversibly attaching themselves to the resin and masking its active groupings from the weaker organic acids. Later experiments with the greater facilities of our own laboratory have given every indication that treating the resin with a strong reducing agent removes these bodies, restoring it to its original capacity. This has been substantiated with test runs on Layton beet juice sent to our laboratory. Indications are that reoccurence of this loss in capacity can be prevented simply and inexpensively.

The question has frequently been raised as to the bacteria growth in the resin beds and their effect on sugar production. After the plant had been in operation some 6 weeks, samples of the resins and various juices and liquors were taken for bacterial study. The following table shows the results of these tests.

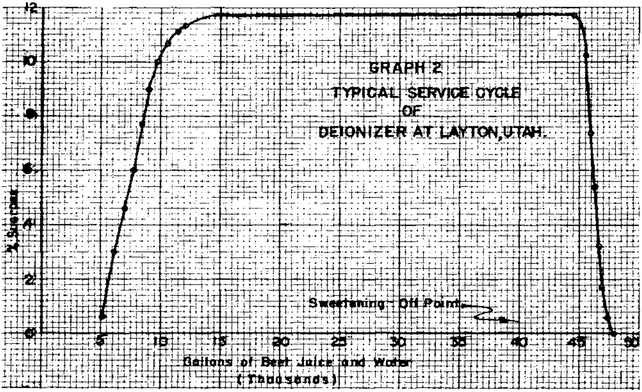
	Flat Sours	Total thermophyles	Total bacteria @ 37°	Molds	Yeast spores
Cation resin end of run		Innumerable	Innumerable	200	-
Anion resin end of run	90	165	Innumerable	125	--
Treated juice first of run		5	620	---	--
Treated juice end of run	---	--	925	---	--
Second carbonation influent to heat exchangers	425	425	22500	10	--
Second carbonation effluent from heat exchangers	2260	2260	Innumerable	---	--
Cation backwash	6250	6250	Innumerable		--
Anion backwash	----	----	210		--

From the above, it is quite evident that while the juice was quite heavily contaminated entering the de-ionization equipment, it was relatively free of objectionable contamination leaving the equipment. Indications are, in this particular case at least, that the resin beds actually acted as a filtering medium to remove some bacteria as well as destroying other bacteria, and further that regeneration cleanses the beds of the accumulated bacteria.

Perhaps the best way to summarize the operation of the plant is to quote a few figures. The following are averages for the entire operating period and in most cases represents the results of several hundred determinations. Brix of influent juice 12.95; brix of effluent juice 11.81; percentage sugar influent juice 12.13; percentage sugar effluent juice 11.61; influent purity 93.76 percent; effluent purity 98.60 percent; pH influent 8.14; pH effluent 7.13; percentage invert influent .13; percentage invert effluent .39.

A study of the percentage sugar figures for influent and effluent juice indicates an overall dilution of 4.28 percent. Graph 2 shows a typical sweetening-on– sweetening-off curve for this plant. It is interesting to note the extreme rapidity with which these steps were accomplished. At Layton, all juice above two brix was sent to the evaporators. Obviously, economics dictates the point to start saving the thin juice and the disposal to be made of the sweet water.

Undoubtedly the next question in your minds is that of sugar losses due to the de-ionization station. The unknown sugar losses at the Layton plant during the time the de-ionization equipment was operating were increased by .24 percent sugar on sugar.



The purity rise figures indicate a non-sugar removal of 77.56 percent. Previous reports of pilot-plant studies have frequently indicated considerably better results. However, the unusually high purity of the untreated juice must be taken into consideration in this case.

A favorable pH condition is indicated by the figures quoted. During the time we were experiencing the resin difficulties previously mentioned, it was necessary to occasionally adjust the pH of the evaporators by the addition of alkali. This required only small dosages, however, due to the unbuffered condition of the juice.

It is believed that the overall picture of .26 percent increase in inversion is quite favorable. However, a considerably lower figure had been expected. As previously indicated some difficulty was experienced with the cooling water supply. About 2 weeks before the end of the campaign, the cold surface water supply failed, necessitating the use of ground water, having a temperature of 66 degrees Fahrenheit. A material increase in the invert was noted following this change.

Reports from the Layton Sugar Company show an extraction of 92.03 percent for the time the de-ionization equipment was in operation. This is a material increase over the approximately 85 percent to be expected on straight house operation, and as reported by Mr. Ellison this morning, they expect to further increase this extraction next campaign by changes to be made in the sugar-end operation.

Final figures on regenerant consumption showed 20.44 pounds of 66 degrees Be' sulfuric acid and 11.81 pounds of anhydrous ammonia used per ton of beets. It is believed that these figures can be materially reduced next campaign.

The success of any new process is dependent upon its ability to improve the product and show a satisfactory economic balance. There is little doubt that the de-ionization process has proved its ability to show an improved product both in appearance and in chemical analysis. Ash determinations on the de-ionized sugar showed results of from .006 to .008 percent as compared to .025 to .035 percent for straight house produced sugar.

The economic balance was not as favorable as originally expected primarily due to the extremely high molasses prices at the present time. One of the major items on the debit side is some 1,400 tons of molasses that were not produced. It is gratifying, however, to note that there still was a substantial savings shown. To these tangible savings can be added such items as no shut-downs for boiling-out of evaporators and pans during the entire campaign, increased efficiencies of the sugar-end operation due to clean heating surfaces and the elimination of the pulling of tubes in the evaporators after campaign. While these are intangible savings on which no dollars and cents value can be placed, they do amount to thousands of dollars per year to a beet-plant operation.