

THE JOURNAL OF AGRICULTURE OF THE UNIVERSITY OF PUERTO RICO

Issued quarterly by the Agricultural Experiment Station of the University of Puerto Rico, for the publication of articles by members of its personnel, or others, dealing with any of the more technical aspects of scientific agriculture in Puerto Rico or the Caribbean Area.

Vol. XLIX

July 1965

No. 3

Effects of Regeneration Procedures on the Performance of Reverse-Cycle Ion Exchange in the Purification of Sugarcane Juices

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INTRODUCTION

The effectiveness of a reverse-cycle arrangement of ion-exchange resins in purifying Puerto Rican sugarcane juices has been described previously (1,2).²

These publications provided data regarding the elimination of various juice impurities and the quality of direct-consumption sugars recovered from the product juices, but did not include information on the chemical requirements for practical regeneration of the exchanger resins. Such data are important for evaluation of ion-exchange processes, since the cost of the regenerating chemicals markedly affects the cost of the process.

An ion-exchange resin used in laboratory experiments is usually treated with several times the stoichiometric amount of regenerating chemical in order to insure complete removal of exhausting ions from the resin. In commercial practice such large excesses of regenerant are not employed for economic reasons, and some exhausting ions remain on the exchanger resin, the quantity varying inversely with the amount of regenerating chemical used (3). The presence of such undesired ions in the resin following regenera-

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² Italic numbers in parentheses refer to Literature Cited, pp. 285-6.

tion effectively reduces the resin capacity in subsequent use, but a compromise between the quality of the product and the quantity of regenerant employed has been accepted as a practical necessity in industrial processing.

A procedure has been described which permitted usage of a large excess of NaOH in regeneration of the first anion exchanger of the reverse-cycle system, and reusing that portion of the waste regenerant which contained a significant concentration of alkali (4). By this means sufficient alkali was provided to assure a superior regeneration of the exchanger with very little wastage of the regenerant. Reexamination of this procedure on a larger semipilot-plant scale was indicated in order to evaluate its effect on exchanger capacity and product quality.

A similar procedure employing a large excess of acid for regeneration of the cation exchanger and reusing the acidic waste regenerant to reduce acid consumption was reported by the same authors (4). They used HCl rather than H_2SO_4 and thereby avoided the difficulties with $CaSO_4$ precipitation in the exchanger bed which occurs during H_2SO_4 regeneration of cation resins containing large quantities of the calcium ion. As H_2SO_4 costs less than half as much on the gram-equivalent weight basis as HCl, exploration of cation-resin regeneration by the cheaper acid seemed desirable.

EQUIPMENT AND MATERIALS

SEMPILOT PLANT

Data from these larger scale tests have been divided into four groups in the text and tables 1 and 2 for convenience. These groups were established according to the mill source of the feed juices used, but from examination of the tables it will be apparent that this arrangement also provided uniformity of the equipment employed, and of regeneration procedures throughout each group of experiments.

Mills A, B, and C are raw-sugar centrals, and the feed juices obtained from these sources were factory-clarified juices. Mill D was the cane-sampling mill of the Agricultural Experiment Station at Río Piedras, and the raw juices obtained from this source were clarified at the Food Technology Laboratory of the Agricultural Experiment Station, as described earlier (2).

The semipilot-plant equipment of the Food Technology Laboratory used for mills A and B tests consisted of three 6-inch-diameter exchanger columns and auxiliary equipment described previously (1). In subsequent tests with juices from mills C and D, these columns were increased in height, the piping and valve assemblies were reorganized, and provision was made for control of the rate of juice flow through the series of exchanger beds by means of individually controlled air-pressure domes as described earlier (2). An industrial-type indicating pH-meter with electrodes con-

TABLE 1.—*Regeneration procedures used in semipilot-plant tests*¹

Regeneration procedure	Data for source of feed juices indicated—			
	Mill A	Mill B	Mill C	Mill D
1st anion resin re- generation: Alkali Regeneration procedure	NaOH Conventional	NaOH Conventional	NaOH Conventional	NaOH Recycling
Cation-resin re- generation: Acid Regeneration procedure	HCl Conventional	HCl Conventional	H ₂ SO ₄ Conventional	H ₂ SO ₄ Conventional
2nd anion-resin re- generation: Alkali Regeneration procedure	NaOH Conventional	NaOH Conventional	NaOH Conventional	NaOH Conventional

¹ Conventional regeneration employs total volume of regenerant as fresh material with no recycling. Recycling procedure is described in the text.

TABLE 2.—*Percentage elimination of juice impurities during semipilot-plant ion-exchange processing*

Item	Source of feed juices ¹			
	Mill A ² (6)	Mill B ² (9)	Mill C (20)	Mill D ² (20)
Carbonate ash	93.9	94.7	91.0	93.1
Protein	88.1	85.9	79.5	74.4
Color	—	—	80.0	93.0
Turbidity	—	—	94.0	81.0
Purity rise	6.9	6.1	3.7	3.7

¹ Numerals in parentheses indicate tests averaged.

² Data reported previously (1). Color and turbidity determined on different basis and values are not comparable with later data.

³ Data reported previously (2).

tained in a pressure-type cell was installed in the regenerant waste line below the first anion-resin bed to permit study of the regeneration of this exchanger resin.

The series of three columns of ion-exchange resins was connected in a reverse-cycle arrangement. In the tests with juices from mills A and B, approximately 0.5 cubic foot of the highly basic anion resin IRA-410 was

contained in the first column, 0.3 of the weakly acidic cation resin IRC-50 in the second, and 0.5 of a mixture of the highly basic anion resin IRA-401S and the weakly basic anion resin A-2 in the third.³

The same exchanger resins were employed in the same order for later tests with juices from mills C and D, but the volumes of resins in the three columns were 0.7, 0.3, and 0.5 cubic foot, respectively.

LABORATORY TESTS

Small-scale ion-exchange studies were conducted with a series of three 2½-inch outside-diameter glass columns approximately 4 feet in length to contain the exchanger resins, and corrosion-resistant pumps, tubing, valves, and tanks of an appropriate size for treating approximately 5½ gallons of juice at the average rate of 2 gallons per hour. A 30-ml.-capacity glass cell designed for use with a laboratory-type indicating pH-meter was used to follow the course of regeneration of the cation column. The electrodes were supported loosely in the open end of this cell, and the regenerant waste flow from the cation bed entered the bottom of the cell, covering the tips of the electrodes before discharging through a side drain into a collecting vessel. The laboratory exchanger columns contained the same resins that were used in the small pilot plant, arranged in the same order. Volumes of approximately 0.08, 0.05, and 0.06 cubic foot of resin, respectively, were contained in the first, second, and third columns.

One lot of clarified-juice syrup from the evaporators of Central Canovanas was stored under refrigeration and diluted to juice density as required to provide feed juices for the small-scale tests.

Both semipilot-plant and laboratory-scale ion-exchange experiments simulated the operation of industrial installations as far as possible. Each step of the process, from sweetening-on, deionization of the juice, sweetening-off, rinsing juice from the system, backwashing of the beds, and regeneration of two of the exchanger beds, was carried out uniformly so that the effects of the regeneration procedures used for the remaining exchanger resin might be detected. The size and construction of the laboratory facilities precluded exact duplication of all semipilot-plant techniques in the smaller equipment, so that the procedures employed for each scale of testing will be described separately.

³ Ion-exchange resins IRA-401S, IRA-410, and IRC-50 were provided by Rohm & Haas Co., Philadelphia, Pa., and the A-2 resin was obtained from Chemical Process Co., Redwood City, Calif. The mention of these products does not mean that they are endorsed by the Puerto Rico Agricultural Experiment Station or the USDA as superior to similar products not mentioned.

PROCEDURES

SEMPILOT-PLANT TESTS

The cold feed juices were warmed to approximately 50° C. in passage to the ion-exchange system, and were passed through the series of exchangers at a rate of from 10 to 15 gallons per hour. When the conductivity of the product flow from the last bed reached 250 μ mho, juice feed was stopped and water passed through the system to rinse out the juice. The water rinse was terminated when a reading of 0 percent refractometric dry solids was obtained on the flow from the last exchanger. All juice from each experiment was collected, mixed, and sampled before discard.

Each bed was then backwashed individually by upward flow of water to reclassify the resin particles, and to wash out suspended materials and traces of sugars. Backwashing of the cation and the second anion columns was attained simply, but waterflow could not remove the large quantities of insoluble matter collected in the first anion bed without prior agitation of this hardened material. Air from jets located at the upper surface of the resin-bed support media supplied sufficient agitation to dislodge this sediment from the partly suspended resin, and when the airflow was stopped, backwash water rinsed the insoluble materials from the bed effectively.

As may be noted in table 1, the resin beds were regenerated either according to a conventional or a recycling procedure in all groups of tests. In the conventional procedure all of the regenerating chemical was applied to the resin bed as fresh material, and the waste product was not fractionated or reused. When conventional procedures were employed during regeneration tests with mill-C or -D juices, the first anion bed was treated with 15 liters of 2 *N* NaOH, the cation resin with 70 liters of 0.2 *N* H₂SO₄, and the second anion resin with 3 liters of 2 *N* NaOH. Experiments with mill-A and -B juices required somewhat smaller quantities of regenerant chemicals for the smaller resin volumes and smaller quantities of juice processed per test. An average of 14 liters of 2 *N* NaOH was used for the first anion resin, 12 liters of 1.25 *N* HCl for the cation, and 2 liters of 2 *N* NaOH for the second anion bed. These quantities of chemicals supplied sufficient excesses to assure good resin capacities during subsequent use. Excess regenerants were rinsed from the beds with water. Rinsing was continued until the free acid or alkali content of the runnings from the bed did not exceed 0.001 *N*, as determined by titration of catch samples.

The recycling procedure for regeneration of the reverse-cycle first anion bed followed a method described by Fort and Smith (4) for reducing the quantity of chemical required for regeneration of this type of exchanger resin. In such a procedure the waste-regenerant flow was separated into fractions according to the concentration of free alkali, and those fractions

with usable concentrations were saved for reuse in a later test prior to fresh alkali.

An average dosage of 38-gm.-eq. weights of NaOH were used for each regeneration of the first anion bed during the mill-D tests. A portion of this was the material saved from a previous experiment and the remainder was fresh 2 *N* NaOH. Water was used to rinse excess regenerant from the bed to a level of approximately 0.001 *N* free alkali on the runnings.

The flow of waste regenerant from the anion bed was separated into three fractions. The first portion was collected until the free alkali strength of the flow from the column increased to 0.5 *N*; the second or alkaline portion followed the first and was collected until the free basicity of the flow decreased to 0.5 *N*; the third or dilute portion followed the second and was terminated when the excess alkali was rinsed from the bed. The fractionation points were determined by titration of catch samples of the flow, or in some instances according to the pH values which coincided with the desired levels of free alkali.

Each waste-regenerant fraction was measured, mixed, sampled, and its content of free NaOH determined. The first fraction was discarded. The second fraction was saved to supply a portion of the regenerating NaOH in a subsequent test. The average free alkalinity in the third fraction was too low for use alone, but was valueless in making up fresh regenerant.

LABORATORY TESTS

Passage of feed juices through the series of laboratory columns was similar to semipilot-plant procedures. No pressure was applied to these columns, and the average flow rate of 1.9 gallons per hour represented the maximum gravity flow obtained with this system. The quantity of juice required to provide a 250- μ mho cutoff point was established experimentally for each series of tests, and this amount was used in all tests made under similar conditions of regeneration. Water rinsing of the system, and collecting, mixing, and sampling of the product juices were done as in the larger scale procedures.

Backwashing of the cation and second anion beds was achieved satisfactorily with an upward flow of water. Backwashing of the first anion exchanger was subject to problems similar to those experienced with the corresponding semipilot-plant column, but the installation of suitable air jets in the smaller glass columns was not feasible. The procedure used in separating the sediment from the resin beads and removing it from the bed included careful control of the backwash waterflow to provide minimum bed expansion and, simultaneously, a vigorous stirring of the bed from above with a long steel rod. This was not a continuous operation, as the column soon filled with water, suspended resin, and sediment, and the flow

of water was stopped until the resin settled. The sediment was lighter than the exchanger resin and remained partly suspended after the resin settled, and backwash water could then flush out any sediment which had been separated from the exchanger beads. The entire procedure was repeated until the quantity of sediment which separated was very small.

The two laboratory anion-exchangers were regenerated conventionally, the first with 2 liters of 2 *N* NaOH and the second with 1 liter of 1 *N* NaOH. Excess alkali was rinsed from the beds with water to a level of 0.001 *N*, free alkali on the runnings. The cation resin was regenerated by one of four schemes: 1, With 1.5 to 2.5 liters of 2 *N* HCl; 2, with 11 liters of 0.2 *N* H₂SO₄ followed by 1 liter of 1 *N* HCl; 3, with 11 liters of 0.2 *N* H₂SO₄ only; 4, with an average of 7.7 liters of once-used H₂SO₄, 0.125 *N* in free acid, followed by an average of 6.2 liters of fresh 0.2 *N* H₂SO₄.

The waste-regenerant flow from the cation exchanger was divided into two fractions in the first, third, and fourth groups of experiments shown in table 3, and into three fractions in the second group of experiments. The end of the first effluent fraction was established by the sharp increase in free acidity of the flow, and the end of the second fraction was determined when the free acidity of the effluent decreased to approximately 0.001 *N*. In the second group of tests all regenerant waste from the HCl treatment was collected as the third fraction. Detection of the end of the first fraction by a rapid decrease in pH from approximately 5 to 1.3 or 1.4 was established during these tests.

All waste-regenerant fractions were collected separately, measured, mixed, and sampled. In the fourth group of experiments the acidic second fraction was saved and recycled in a subsequent experiment prior to fresh acid. An average dosage of 2.2 gm.-eq. weights of H₂SO₄ was used in regeneration of the cation exchanger in these recycling tests, this amount representing the sum of the once-used acid and fresh 0.2 *N* acid.

ANALYTICAL METHODS

Analyses of sugar-containing materials followed procedures used in the cane-sugar industry, including solids by low-temperature vacuum-oven drying on sand, carbonate ash after incineration at 550° C, reducing substances by the Lane-Eynon method, protein as Kjeldahl nitrogen \times 6.25, true purity based on chemical determination of sucrose by Lane-Eynon measurement of invert sugars before and after enzymatic inversion of sucrose, and turbidity and color after the method of Balch (5).

Analyses of the four principal juice cations in some regenerant wastes were made on aliquots of these materials after evaporation and thorough oxidation with HNO₃ and HClO₄. Sodium and potassium were determined by flame photometry, calcium as the oxalate, and total calcium and magne-

sium by EDTA titration. Values for magnesium were obtained by difference. Similar procedures with oxidized carbonate ash samples provided values for these same cations in feed and product juices.

RESULTS AND DISCUSSION

SEMPILOT-PLANT TESTS

The free alkalinity of the waste regenerant flowing from the first anion resin increased during the early stages of regeneration to a maximum of approximately 2 *N*, then decreased as a result of dilution by the rinse water. The first fraction of these runnings contained most of the exhausting anions

TABLE 3.—Laboratory data on cation regeneration

Regenerating acid	Acid normality	Acid dosage per test	Tests averaged	Feed-juice ash processed in test	Removal of				Product-juice ash (dry solids)	Feed-juice cation remaining in effluent			
					Ash	Protein	Color	Turbidity		K	Ca	Mg	Na
		Gm. eq. wt.	Number	Pounds	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
HCl	2.0	4.82	7	0.187	94.1	85.5	84.4	82.1	0.134	6.1	4.6	0.7	14.6
H ₂ SO ₄ and HCl	.2 1.0	2.20 1.05	3	.175	93.1	83.3	84.2	80.1	.174	7.5	4.3	5.5	13.2
H ₂ SO ₄	.2	2.23	4	.179	89.9	69.8	85.3	81.6	.240	11.1	4.2	4.0	17.4
H ₂ SO ₄ and H ₂ SO ₄	.126 .2	.96 1.24	5	.169	89.4	71.4	83.1	80.6	.251	10.2	5.1	1.5	19.2

which the resin had removed from the cane juice and which the NaOH subsequently regenerated from the resin bed. It also contained considerable reddish-black juice-coloring materials retained by the resin, and had an average strength of 0.2 *N* free NaOH. This fraction was discarded because of its low concentration of free alkali and high concentration of objectionable color. The second fraction was collected so that it would contain all material with a free-alkali strength in excess of 0.5 *N*. Some salts and coloring materials were also present, but the quantities were not so great as in the previous fraction, and its average free-alkali strength of 1.23 *N* was sufficient to justify its reuse for regeneration of the resin in a subsequent experiment. The third fraction taken was small in volume, colorless, with an average of 0.05-*N* free-alkali strength. Its use in preparing fresh alkali for the next experiment made use of its NaOH content.

Fractionation of the waste regenerant from the first anion bed into three portions and reusing the second and third fractions resulted in the loss of only that free alkali which was contained in the first fraction. The average loss of NaOH was 1.96 gm.-eq. weights per test for an average stoichiometric requirement of 13.91 gm.-eq. weights of alkali neutralized in the regeneration process, a wastage of 14.2 percent of the theoretical amount of NaOH necessary. The consumption of 114.2 percent of the stoichiometric quantity of NaOH required to regenerate the first anion resin agrees well with the 110 percent reported by Fort and Smith (4).

A few tests were made to determine the regenerant fractionation points by the pH of the effluent flow. The desired pH values were not established precisely, but the ranges found were from 7.3 to 7.6 for termination of the first fraction and 11.2 to 11.6 for the end of the second fraction.

No significant decrease in capacity of the first anion resin was noted in the tests where waste NaOH regenerants were reused. In table 2 no relationship is noted in the percentage eliminations of juice impurities and the anion regeneration practices employed. The apparent improvement in turbidity eliminations in mill-C tests as contrasted to mill-D tests doubtless resulted from the superior filtration effect provided by the greater quantities of suspended matter in these juices from mill C.

LABORATORY TESTS

The one lot of syrup which provided all the feed juices changed very little in analysis during the period of experimentation, so that the data presented in table 3 represent the effect of regeneration procedure on the product quality without need of allowing for the effect of changes in feed composition. The average mineral composition of the feed juice was determined in percentage as 1.148 of K_2CO_3 , 0.625 of $CaCO_3$, 0.157 of MgO , and 0.089 of Na_2CO_3 , on the juice dry-solids basis. No analyses were made of the sucrose or invert sugar contents of these feed or product juices.

The average of 4.82 gm.-eq. weights of HCl employed for regeneration of the cation resin in the first group of tests in table 3 represented an excess of approximately three times the stoichiometric quantity of acid required. Data from these tests were included to serve as examples of the best performance to be expected from the system under the experimental procedures used. Earlier studies had indicated that reuse of HCl made such large dosages of this acid practical, since adequate regeneration was attained with a consumption of 105 percent of the stoichiometric acid requirement (4). The percentages of the various cations which remained in the effluent juices, as shown in table 3 where HCl was the regenerant, probably resulted from leakages during late stages of juice deionization and during rinsing of juice from the exchanger bed. In later groups of experiments these figures

include leakage of feed-juice cations, as well as some cation elution which resulted from incomplete resin regeneration.

The sulfuric acid used in these regenerations did not exceed 0.2 *N* in strength, since this concentration was found to be the maximum where CaSO_4 did not precipitate in the cation bed. The dosage of H_2SO_4 used to regenerate the exchanger in the last three groups of tests represented slightly more than double the stoichiometric amount required. In the second group of tests, H_2SO_4 regeneration of the cation bed was followed by a regeneration with HCl to obtain information regarding the cations which the former acid did not remove from the exchanger.

Evidence of the superior performance of the resin following HCl regeneration was noted in the greater weights of feed ash processed, the greater percentage eliminations of impurities, and, most clearly of all, in the lower ash-percent-solids of the effluent juices as presented in table 3. Color and turbidity changes apparently were independent of the regeneration procedure. The ash-percent-solids of the effluents increased by 79 with simple H_2SO_4 regeneration, 29 when H_2SO_4 was followed by an HCl rinse, and by 87 when H_2SO_4 was recycled. Examination of the cation compositions of the effluent juices reveals that potassium had primary responsibility for all of these mineral increase, with some contributions due to magnesium and sodium ion increases.

Further evidence of the superiority of HCl over H_2SO_4 as cation regenerant is available in earlier data obtained from reverse-cycle tests presented in table 2. HCl regeneration in mills A and B tests provided purity rises of 6.1 and 6.9 points, while the mill-C and -D tests with H_2SO_4 as the regenerant resulted in 3.7-point purity rises. In this table lower ash and protein eliminations coincided with smaller purity increases, suggesting that incomplete regeneration of the cation resin by H_2SO_4 was responsible for failures to obtain maximum purity increases.

The calcium content of the effluents noted in table 3 was remarkably uniform, increasing only when H_2SO_4 was recycled in the fourth group of tests. Significant quantities of calcium and magnesium were found in the waste of the HCl rinse in the second group of tests, and since the levels in the effluents were not increased in the third group where no HCl rinse was used, it may be assumed that, with H_2SO_4 regeneration, these cations tend to accumulate in the resin. Sodium contents did not change appreciably from group to group, though there was an increase where H_2SO_4 was the sole regenerant. H_2SO_4 at approximately one-third the cost of HCl , on the basis of chemical equivalent weights of the active materials in the commercial grades of acids, would be more economical for regeneration of the resin, provided this acid satisfactorily removed calcium and magnesium ions.

Significant quantities of magnesium and calcium were retained on the first anion exchanger of the reverse-cycle system during passage of cane juices through this highly basic resin. This exchanger liberated the bases from the juice minerals, and the insoluble hydroxides of calcium and magnesium which formed were precipitated in the bed, and remained there until the resin was backwashed, at which time they were rinsed out as waste. The quantities of these cations precipitated in the anion resin bed in this manner could be determined by analysis of the backwash wastes, provided the backwashing procedure effectively removed all of such materials from the resin bed. Unfortunately, backwashing of the small laboratory column was neither as efficient nor as uniform as that of the semipilot-plant bed, and complete removal of the insoluble hydroxides could not be assured. Analyses of the backwash wastes from the first anion bed were not available from all tests, but as much as 40 percent of the magnesium and 5 percent of the calcium in the feed juices were accounted for in some of these materials. Following special treatment of this anion bed with HCl, the first and second tests provided backwash wastes with 4 and 14 percent, respectively, of the feed magnesium, but there was no significant change in the calcium content. Available data are inadequate to explain this marked decrease in the magnesium content of the backwash following elimination of this ion from the resin by HCl treatment, but it seems probable that efficient backwashing may reduce but not eliminate a buildup of magnesium ions in this anion bed.

Inadequate backwashing of the first anion bed may result in a buildup of the insoluble $Mg(OH)_2$ in this exchanger and a partial blockage of the resin. Smaller quantities of $Ca(OH)_2$ would be similarly involved in these residues. Some of these materials might be leached by subsequent juices, but the quantities would be small, as the high pH of the juices leaving this bed would not favor formation of their soluble salts. The retention of calcium and magnesium ion in the cation exchanger following H_2SO_4 regeneration would exert a greater effect in increasing the ash of product juices, though this effect would not necessarily be indicated by an increase of these two cations in the effluents. This type of weakly acidic cation exchanger exhibits a marked preference for calcium and magnesium over potassium and sodium, so that the presence of calcium and magnesium on the resin following regeneration would be detected by an increased potassium and sodium ion leakage into subsequent lots of juice rather than by increased displacement of calcium and magnesium ions from the resin.

No explanation can be offered for the leakage of rather large quantities of calcium and sodium ions into the effluent juices of the first group of tests, or for the decrease in potassium and magnesium contents of the effluents in the fourth group of tests.

SUMMARY

Investigations of the chemical requirements for regeneration of the first-anion and the cation-exchanger beds of the reverse-cycle system were undertaken in these studies, but no attempt was made to establish the requirements for the second-anion exchanger bed. A large dosage of NaOH to provide superior exchanger capacity was used for the highly basic anion resin in first position, and by recycling usable portions of the waste regenerant in a subsequent test, an average of 114 percent of the stoichiometric quantity of NaOH was consumed per test. No change in performance of the exchanger system was detected following adoption of such a regenerant recycling procedure.

Efforts to obtain satisfactory regeneration of the weakly acid cation exchanger with H_2SO_4 were discouraging. The quantity of material which could be processed per test was less, the percentage removals of ash and protein were less, and the ash-percent-dry solids remaining in the product juices was 79-percent greater than when HCl was employed as regenerant. Juice purities following ion-exchange processing were found to have increased by approximately one-half as much with H_2SO_4 regeneration as with HCl. Examination of the product juices revealed that their potassium-ion contents were much greater during H_2SO_4 regeneration tests than while HCl was used. Apparently the large quantities of calcium and magnesium ions found in cane juices were not efficiently regenerated from the exchanger resin by H_2SO_4 , and that part which remained in the resin during subsequent juice deionization increased the leakage of potassium ions into the product juices. Although HCl was much more expensive than H_2SO_4 , the recycling of excess regenerant to reduce the quantity consumed to approximately 105 percent of the necessary stoichiometric requirement would make the use of this more costly acid economically attractive.

Information was obtained in these tests which showed the importance of efficient backwashing of the highly basic first anion bed in order to eliminate significant quantities of $Mg(OH)_2$, and organic materials precipitated on the resin particles during passage of juices through the bed. The effective backwashing obtained in the semipilot-plant column was not always attained in the smaller laboratory column and some insoluble materials remained following backwashing and regeneration.

RESUMEN

Se llevó a cabo un estudio de las técnicas más adecuadas para regenerar las resinas aniónicas y catiónicas que se usan en la purificación del jugo de caña clarificado cuando se utiliza el ciclo invertido. Bajo este método, el jugo se trata primeramente con la resina aniónica y luego con la catiónica.

Para lograr una regeneración efectiva de la resina aniónica se usó una

cantidad relativamente alta de NaOH, pero como fue posible utilizar de nuevo parte de la solución regeneradora ya usada, en la regeneración de las resinas en ciclos subsiguientes, la cantidad promedio de NaOH que se usó por ciclo se redujo a 114 por ciento de la cantidad estequiométrica requerida. No se observó pérdida en la capacidad regeneradora de la resina aniónica por el uso repetido de la solución.

No se logró que la resina catiónica actuara eficientemente cuando se regeneró con ácido sulfúrico. Cuando se usó este ácido como regenerador, la cantidad de jugo que pudo tratarse por ciclo fue menor que cuando se usó el ácido clorhídrico. Fue menor el porcentaje de cenizas y proteínas removidas por ciclo, pero más alto el contenido de cenizas en el jugo que se trató. Cuando se usó el ácido clorhídrico en la regeneración de la resina catiónica, se observó un aumento en la pureza del jugo, doble al que tuvo lugar al regenerarse con ácido sulfúrico.

Los jugos que se trataron con las resinas catiónicas regeneradas con ácido sulfúrico tenían un contenido mayor del ion de potasio que los que se trataron con resinas regeneradas con ácido clorhídrico. Aparentemente, la regeneración con el ácido sulfúrico no removió con efectividad los iones de potasio y magnesio. Cuando se trataron los jugos nuevamente con estas resinas, parte de los iones de potasio pasaron a los jugos, aumentando la concentración de este ion en el jugo tratado.

Fue posible reducir la cantidad de ácido clorhídrico necesaria para regenerar las resinas, a 105 por ciento de la cantidad estequiométrica, usando en ciclos subsiguientes parte de la solución del ácido que se usó en regeneraciones anteriores. Aunque el ácido clorhídrico es más costoso que el sulfúrico, la reducción de la cantidad necesaria para la regeneración a los niveles indicados, hace factible su uso en la regeneración de las resinas en operaciones industriales.

Estas pruebas han demostrado la importancia de lavar adecuadamente la resina aniónica de la primera columna para remover el precipitado de $Mg(OH)_2$ y de materia orgánica que dejan los jugos en su paso por las columnas. En las pruebas llevadas a cabo en escala semi-experimental pudieron lavarse las resinas adecuadamente, pero no se pudo lograr en las pequeñas columnas que se usaron para las pruebas de laboratorio.

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