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ACID/SUGAR SEPARATION USING ION EXCLUSION RESINS: A PROCESS ANALYSIS AND DESIGN

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ABSTRACT

The economic feasibility of using acid hydrolysis to convert agricultural waste products, such as corn stovers, to alcohol is very dependent upon developing a cost effective sulfuric acid-glucose sugar separation process. Presently, an acid/sugar stream from sulfuric acid hydrolysis of cellulose is treated with lime to precipitate the acid as gypsum. This technique not only consumes acid and lime, but also generates waste gypsum that is sent to a landfill. An ion exclusion process to perform acid/sugar separation has been designed using standard resins. Economic analysis based on a process computer model indicates that ion exclusion is 40 % less costly than a lime precipitation process.

INTRODUCTION

During the oil crisis of the last decade, the necessity of finding a fuel substitute for gasoline became apparent. Alcohol (ethanol) was mixed with gasoline. The gasohol mixture not only preserves crude oil resources, but reduces combustion pollution (1). To meet the expected future alcohol demand of the United States, the National Fertilizer Development Corporation (NFDC) developed several processes which uses naturally available cellulosic materials to produce alcohol. Two major hydrolysis processes were simultaneously developed which use sulfuric acid to convert cellulosic raw materials into an acid/sugar mixture (2). Acid was separated from glucose sugar by lime precipitation. The sugar was fermentated in a later operation to yield alcohol. Because acid was not recovered and waste gypsum produced, the economic feasibility of both hydrolysis processes was handicapped.

Several separation processes were investigated as an alternative to precipitation. However, competing processes based on activated charcoal adsorption and solvent extraction were judged not to be economically sound (3). In contrast, separation by ion exclusion (IE) was favored because acid can be recovered and reused. It is the purpose of this work to define an ion exclusion acid/sugar separation process and to project its economic feasibility.

Proposed Ion Exclusion Process Description

An ion exclusion resin can divide an acid/sugar mixture into its components. Sulfuric acid is excluded from entering the porous resin because of ion repulsion due to a high chemical potential of acid groups inside the resin. Thus, acid will pass through a resin-packed bed faster than sugar which penetrates the resin's microporous structure. When an acid/sugar mixture is passed through a resin bed, three fluids will exit at different elution times. An acid-rich stream will elute first, followed by a dilute acid/sugar interface stream and then a sugar-rich stream. Evaporation of the acid-rich and sugar-rich streams gives the desired acid and sugar concentrations required for hydrolysis and fermentation. The dilute acid/sugar interface stream can be recycled. The steam generated in the evaporation steps can be condensed to improve heat recovery and used as the fluid to carry the acid/sugar feed through the packed column.

PREVIOUS WORK

Wheaton and Bauman (4) investigated the performance of IE columns using several resins and both organic and inorganic mixtures. A later study not only quantified the degree of separation by IE, but also investigated the separation of two or more non-ionic components (5). Simpson and Bauman concluded that the major operating costs involved in IE are for power and water (6). After establishing the physical possibility of IE, Bauman et al. developed the procedures needed to design IE columns (7). Ion exclusion techniques have been used for separations involving ethylene glycol (8) or glycerine (9-13). The commercial feasibility of sugar juices purification by IE was also investigated (14). Ion retardation studies related to the separation of electrolytes from non-electrolytes (sodium chloride from sucrose) using packed columns have also been reported (15).

Ladisch et al. investigated the separation of sulfuric acid from glucose by IE using Rohm and Haas IR 118 H^+ resin (16). A mathematical model based on axial dispersion was used to analyze the process behavior. Their studies indicated that the IE process exhibited high feasibility for acid-sugar separation. However, no economic analysis was performed to estimate commercial feasibility.

Mathematical models have been proposed to predict the IE process performance for a given system and set of operating conditions. Huang et al. (17) compared radial flow to vertical flow chromatography. An analysis of the IE process by an equilibrium plate model with non-linear sorption isotherms was reported by Wheelock (18).

SEPARATION THEORY

An IE packed column volume consists of three parts (6): [1] macropore, also called void or interstitial volume, V_o, the liquid volume between the resin beads; [2] micropore, also known as occluded volume, V_p, the liquid volume held within the resin; and [3] the crosslinked polymer resin network volume, V_r. This distribution coefficient, or the partition coefficient, K_d, of a solute is the ratio of the species concentration in the micropore volume to its concentration in the macropore volume.

The K_d value varies with the nature of the solute (ionic, non-ionic), nature of the resin (acidic, basic, resin crosslinkage density, particle mesh-size), solution composition, and temperature. The difference in K_d values for different solutes is a measure of the ease of separation achievable by ion exclusion.

The K_d value also gives the average time a solute will elute from a packed resin bed. The fraction of resin volume penetrated by a solute is proportional to the ratio of solute concentration inside the resin to the concentration outside the resin. Thus, the elution volume or the total fluid volume experienced by a species V_e is

$$V_e = V_o + K_d V_p \qquad (1)$$

Solutes which have different partition coefficients are separated in their passage through an IE column. The time that a species elutes from a column is proportional to its elution volume.

Process Mathematical Model

A finite length equilibrium technique was used to perform a stage-by-stage IE column material balance (19). A single solute material balance at volumetric flow rate, Q, over the nth column stage of height, H, and column cross sectional area, S, gives the following for a resin bed having α and β macropore and micropore volume fractions, respectively.

$$QC_{on-1} - QC_{on} = HS\alpha(dC_{on}/dt) + HS\beta(dC_{pn}/dt) \qquad (2)$$

where C_{on-1} = solute concentration in the macropore phase at the n-1 stage
C_{on} = solute concentration in the macropore phase at the nth stage
C_{pn} = solute concentration in the micropore phase at the nth stage

The solute mass gained or lost by advancing one stage down the column is equal to the total solute mass existing in equilibrium between the macropore and micropore phases of a preceding stage. The distribution coefficient can be used to relate the equilibrium concentration of a solute in the micropore phase to its concentration in the macropore phase.

$$C_p = K_d C_o \qquad (3)$$

Combining Equations 2 and 3 and using finite differences to approximate the derivatives, a final working equation over length increment, h, and time increment, k, can be obtained (19).

$$\psi_{n,s} = [\psi_{n-1,s} + (hK_{\alpha\beta}/k)\psi_{n,s-1}](1 + hK_{\alpha\beta}/k) \qquad (4)$$

where $\psi_{n,s}$ = dimensionless concentration of solute in macropore phase at stage n and time increment position s
$\psi_{n-1,s}$ = dimensionless concentration of solute in macropore phase at stage n-1 and time increment position s
$\psi_{n,s-1}$ = dimensionless concentration of solute in macropore phase at stage n and time increment position s-1
$K_{\alpha\beta}$ = $(\alpha+\beta K_d)/(\alpha+\beta)$

An original FORTRAN program based on Equation 4 was modified and converted into Turbo Pascal and used to simulate the IE process. Input program parameters are the macropore volume fraction, α, micropore volume fraction, β, distribution coefficient of the solute, K_d, incremental time, k, total number of stages, N, volume fraction of the empty column used as feed, ν, and the dimensionless time at which to end the program. The program predicts the dimensionless concentration, ψ, of a species in the eluent leaving the column as a function of dimensionless time, τ. Thus, an elution profile is generated.

The above model was developed for a single component system. It was used to simulate column performance on an aqueous feed of only acid and then used again for an aqueous feed of only sugar. Thus, interaction between components was not accounted for in the model. Input parameters to the computer model, K_d, and N were varied until the model simulated elution profile matched as closely as possible the profiles generated by Ladisch (16). Since the acid and sugar differed in their K_d values, two height equivalent to a theoretical plate (H.E.T.P.) were obtained. Model simulations for both acid and sugar elution profiles were performed by varying feed volume column loading, ν, from 4.0 to 20.0 % of empty column. For each value of ν, the model-simulated elution profile was compared with a Ladisch experimental profile to obtain K_d and H.E.T.P. values at each process condition. Although the acid component was first assumed to have a K_d value of zero (ideally total exclusion), the model matching of Ladisch's experimental data yielded a value of 0.08.

EXPERIMENTAL

Resin Equilibrium Measurements

All resin samples were soaked in deionized distilled water overnight before equilibrium experiments were performed to determine distribution coefficients. Adhered water was removed by using a syringe or centrifuge. Pre-weighed, water-soaked, resin samples (Rohm and Haas, IR 122 H^+) were equilibrated in aqueous sulfuric acid/glucose solutions of known concentration. The resin-solution mixtures, contained in sealed bottles, were gently agitated in a temperature controlled bath. After two or three hours, equilibrium was reached as indicated by no significant change in the concentration of sugar in the supernatant. Once equilibrium was reached, the supernatant solution was decanted from the resin and the adhered water was removed. Resin and supernatant were then analyzed for composition to determine distribution coefficients.

Sugar Detection

Aqueous solutions containing 8.0 weight % sulfuric acid and α-D glucose of known concentration (1.0 to 20.0 % by weight) were prepared. These solutions were characterized by optical rotation measured with an optical polarimeter (Model SR 6 Polarimeter, PolyScience Corporation). The sulfuric acid present in our solutions caused an almost 50 % decrease in optical rotation values compared to sugar solutions containing no acid. Thus, optical rotation calibration of standard glucose samples required the presence of a known quantity of acid. To prevent error from possible variation in acid content, the sulfuric acid concentration was adjusted to 8.0 % by weight in all supernatant solutions. A linear calibration plot of sugar concentration versus solution optical rotation in 8.0 % acid was obtained. The concentration of sugar in a solution was thereafter determined from an optical rotation measurement and the calibrated curve.

RESULTS AND DISCUSSION

Resin Equilibrium Studies

IR 122 H^+ resin equilibrium data was generated at solution temperatures of 5°, 25°, and 55°C. This is a polystyrene strongly acidic cation exchange resin with 10.0 % crosslinking. The concentration of sugar in the solutions varied from 5.0 to 17.0 % by weight, and the temperature ranged from 5.0 to 55.0°C. The ratio of the weight of the resin to that of the solution was 50.0 %. The moisture present in the resin (62.4 wt. % of bottled resin) was accounted when evaluating sugar concentrations within the resin micropore and in supernatant solutions. Table I shows the effect of temperature and solution concentration (with 8.0 % by weight acid) on the sugar distribution coefficient.

TABLE I

Sugar Distribution Coefficients (K_d)
of IR 122 H^+ Resin

Sugar Concentration	Temperature °C		
Wt. %	5	25	55
5.0	0.48	0.46	0.36
11.0	0.51	0.46	0.34
17.0	0.68	0.56	0.48

IR 122 H^+ resin favors sugar sorption at lower temperatures. However, this temperature dependence is not strong and thus, the additional process expense of fluid cooling is not economical. The values of the distribution coefficient do not exhibit a significant variation with sugar concentration at feed sugar concentration less than 12 %. However, there is an increase in K_d as sugar concentration increases above 12 %.

Comparison Between Model And Experimental Data

The process model was used to estimate IE separation process performance using different resins, flow rates, feed loading schemes, components, and total number of stages. Acid and sugar elution profiles for IR 118 H^+ resin packed in 1.0 inch diameter and 24.0 inch long columns as reported by Ladisch was simulated using the computer model. The shape and area under the model and data profiles were similar (see Figures 1 & 2). However, the elution profiles did not exactly superimpose. At high concentrations, the acid experimental profile is slightly skewed to the right of the model profile. The model elution profile for the sugar component was lower in peak height compared to the experimental profiles. However, the leading and trailing edges of the model and experimental profiles match closely for both components.

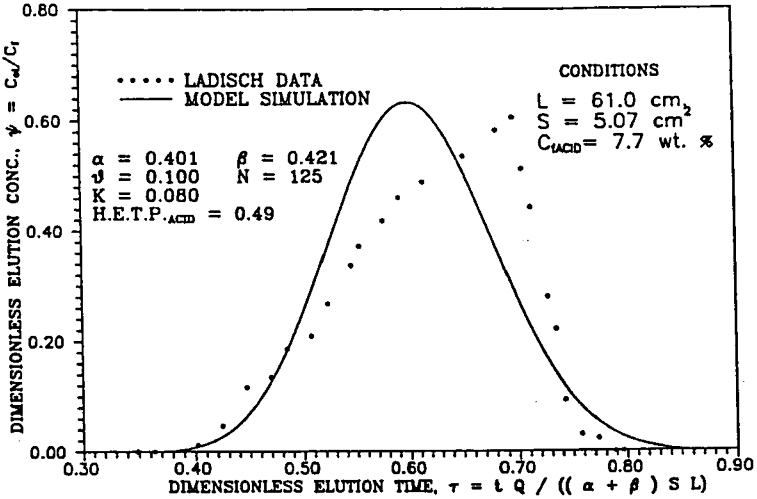


Figure 1. Comparison between model and Ladisch experimental acid elution profiles for IR 118 H^+ resin at 55°C.

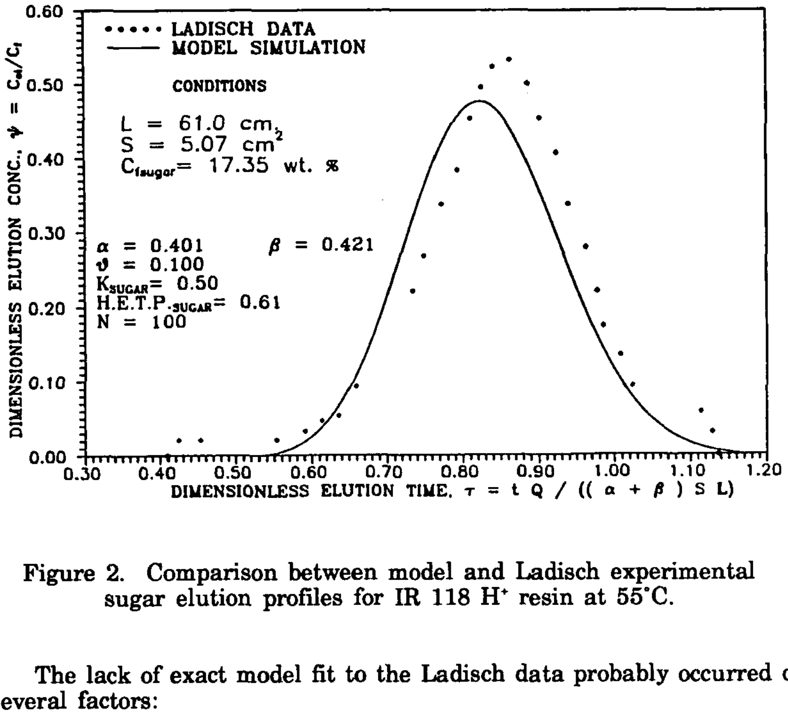


Figure 2. Comparison between model and Ladisch experimental sugar elution profiles for IR 118 H⁺ resin at 55°C.

The lack of exact model fit to the Ladisch data probably occurred due to several factors:

1. Acid and sugar were assigned K_d values of 0.08 and 0.50, respectively. These values are not constant but vary slightly with fluid composition. Thus, K_d values probably vary with column position.

2. The macropore (α) and micropore (β) volume fraction values employed for the resin are not constant. The swelling behavior of the resin varies with the nature of the solution. The resin shrinks with acid present. Thus, α and β values vary with column position.

3. At higher feed values one would expect larger deviations from an equilibrium model. This is indicated by the H.E.T.P. increase observed as the feed volume increased.

4. The model does not correct for variations in fluid buoyancy brought about by separation of components.

Many past experimenters defined feed volume, ν, on the basis of bed macropore volume. However, our definition of ν is based on an empty column volume. Thus, when matching the literature data of with the model simulated profiles we accounted for any differences in the definition of ν. The range of ν from 10 to 50 % in Ladisch's experiments corresponds to a range of 4.0 to 20.0 % in our computations. This ν range falls within values recommended for optimum process performance and has been confirmed by several authors (4,6,11).

The elution time and H.E.T.P. for the acid and sugar components depended on the feed volume. Both increased with an increase in feed volume (see Table II).

TABLE II

Variation of Process Parameters with Fraction Feed Loading

Solute	Parameter	Feed Fraction Loading, ν 0.04	0.10	0.20
Sugar	H.E.T.P. (cm)	0.41	0.61	0.94
	K_d	0.50	0.55	0.45
Acid	H.E.T.P. (cm)	0.10	0.49	1.22
	K_d	0.03	0.08	0.05

Increasing feed volumes without increasing the total number of stages diminished component separation performance. At ν equal to 10.0 %, the model and Ladisch experimental profile closely matched. This was taken as a base case and the H.E.T.P. found at this condition was used for commercial process design and economic analysis.

The number of stages for a commercial process was increased above the base case for two reasons: to lower component dilution and to minimize intermixing of components (6,8). As expected when the number of stages increased, predicted species elution profiles narrowed in widths and simultaneously increased in heights, minimizing cross-contamination of sugar and acid elution profiles. This decreases the quantity of the interface stream which must be recycled.

Process Flowsheets

The flowsheet (Figure 3) and mass-energy balances for the precipitation process were obtained from the NFDC (2,20). The flowsheet developed for the proposed commercial IE process is shown in Figure 4. Table III identifies the different streams shown in Figure 4 (20). A reasonably effective heat recovery scheme has been incorporated into the IE process.

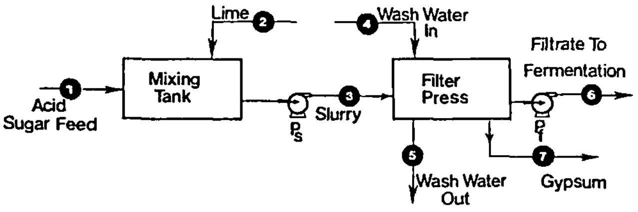
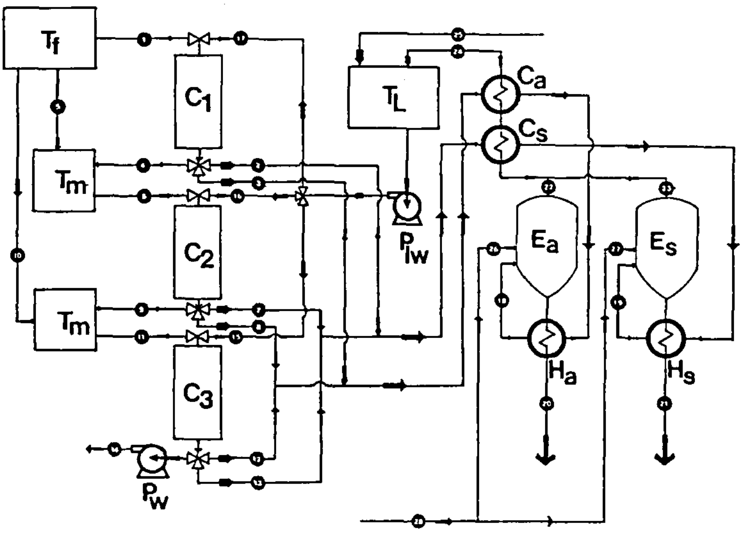


Figure 3. Flowsheet of the precipitation process. P_S = pump for sugar/gypsum slurry stream to filter press, P_f = pump for sugar filtrate stream to fermentation unit.



C_a	acid evaporator steam condenser	H_s	acid free sugar feed pre-heaters
C_s	sugar evaporator steam condenser	P_{LW}	leach water pump
$C_{1,2,3}$	ion exclusion columns	P_W	waste stream disposal pump
E_a	acid evaporator	T_f	acid/sugar mixture feed tank
E_s	sugar evaporator	T_m	acid/sugar mixture tank
H_a	sugar free acid feed pre-heaters	T_L	leach water tank

Figure 4. Flowsheet of the proposed ion exclusion process.

TABLE III

Heat and Mass Balance for Ion Exclusion Process

METHANE GAS (lb/hr)	TEMPERATURE (°F)	DENSITY (lb/ft^3)	VOLUMETRIC FLOW RATE (ft^3/hr)	MASS FLOW RATE (lb/hr)	WATER (lb/hr)	SUGARS (Glucose,Xylose) (lb/hr)	SULFURIC ACID (lb/hr)	STREAM #	Description & Units
	77.7	77.7	92.7	7,207	5,333	1,319	555	1	Acid/Sugar Feed Solution To Unit
	77.7	62.4	100.7	6,284	5,593	475	216	2	Acid/Sugar Eluen From Unit - I
	77.7	77.7	11.8	923	683	169	71	3	Acid/Sugar Make Up Feed To Tank-
	77.7	63.9	168.9	10,795	9,951	844		4	Unit-I Sugar Cut To Sugar Evapn.
	77.7	64.1	123.1	7,895	7,756		339	5	Unit-I Acid Cut To Acid Evapn.
	77.7	62.4	112.4	7,207	6,276	644	287	6	Acid/Sugar Feed Soln. To Unit-II
	77.7	----	--------	------	------	----	----	10	Acid/Sugar Make Up Feed To Tank-
	77.7	62.4	100.7	6,284	5,940	232	112	8	Acid/Sugar Eluen From Unit-II
	77.7	64.1	123.1	7,895	7,756		175	9	Unit-II Acid Cut To Acid Evapn.
	77.7	62.4	173.0	10,795	10,382	412		7	Unit-II Sugar Cu To Sugar Evapn.
	77.7	62.4	100.7	6,284	5,940	232	112	11	Acid/Sugar Feed Soln. To Unit-II
	77.7	62.4	98.1	6,123	5,993	81	48	12	Acid/Sugar Loss From Unit-III
	77.7	62.4	171.7	10.715	10,563	151		13	Unit-III Sugar Cut To Sugar Eva
	77.7	62.4	126.5	7,895	7,831		64	14	Unit-III Acid Cu To Acid Evapn.
	77.7	62.4	295.6	18,447	18,447			15	Leach Water To Unit-III
	77.7	62.4	284.7	17,766	17,766			16	Leach Water To Unit-II
	77.7	62.4	287.9	17,967	17,967			17	Leach Water To Unit-I
	77.7	64.1	373.2	23,921	23,343		578	18	Dilute Acid Feed To Acid Evapn.
	77.7	62.4	517.8	32,303	30,896	1,407		19	Dilute Sugar Feed To Sugar Evapn.
	77.7	114.6	5.2	5,898	12		578	20	Conc. Acid To Cell. Hydrolysis
	77.7	65.8	150.8	9,908	8,501	1,407		21	Conc. Sugar Syru To Fermentation
	212.0	0.042	0.55 x 10^6	23,331	23,331			22	Steam From Acid Evaporator.
	212.0	0.042	0.53 x 10^6	22,395	22,395			23	Steam From Sugar Evaporator.
	77.7	62.4	732.8	45,726	45,726			24	Combined Cooled Condensate Water
	77.7	62.4	135.6	8.464	8,464			25	Make Up Water To Leach Water Tank
1,084	77.7	0.044	24,642	1,084				26	Methane Gas For Acid Evapn.
1,040	77.7	0.044	23,650	1,084				27	Methane Gas For Sugar Evapn.
2,124	77.7	0.044	48,292	2,124				28	Methane Gas Supply Line Feed

The two processes essentially perform the same duty of acid-sugar separation; however, the processes differ in several ways. The precipitation process is very simple and requires less equipment, but produces large quantities of waste gypsum as it consumes acid and lime. The IE process involves more equipment and energy for evaporation units, but produces negligible waste products. The IE process also needs more supervision and operational control because of its inherent operational complexity.

Process Design and Optimization

The IE process was designed using the same sugar output that was achieved by the precipitation process. IE process studies using computer simulation were performed to estimate parameters for optimizing process economics. To accomplish this, several key factors were examined such as maximization of acid/sugar separation, minimization of sugar or acid loss, and use of minimum recycling.

First, to obtain maximum acid/sugar separation, the number of stages were increased using fixed feed volume loading. The Ladisch data indicated an optimum feed loading value of $v = 0.10$. For this loading, the number of stages required for the most economical separation were found to be 500 for sugar and 625 for acid. Figure 5 shows the expected elution profiles at these conditions.

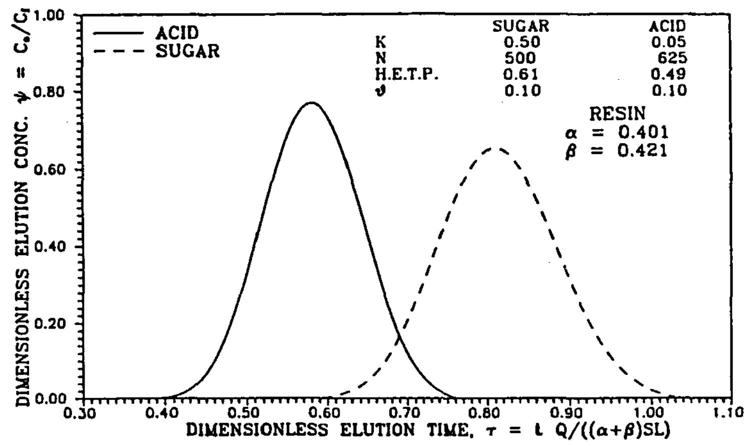


Figure 5. Scale-up of the model simulated acid/sugar elution profiles for IR 118 H^+ resin.

Second, to minimize the amount of eluent recycling required, the use of several columns in series was considered (see Figure 4). Columns were included until the cost of an additional column was more expensive than the value of sugar recovered by adding a column. In this manner, three columns in series were found to be optimum.

Economic Analysis

The operational cost per hour of both the precipitation and proposed IE acid/sugar separation processes producing the same total sugar output were estimated using a spreadsheet analysis (20). Cost based on the value of a 1988 dollar were categorized into chemicals, equipment, power, and labor. A cost comparison of processes is shown in Table IV.

TABLE IV
Comparison of Precipitation with Ion Exclusion

	Process Cost in $/Hour of Operation	
Cost Category	Precipitation	Ion Exclusion
Chemicals	1240	283
Equipment	28	287
Labor	185	167
Power	5	96
Total	1458	832

In the IE process, acid and sugar losses are insignificant. Also, water which is 20 to 27 % of empty column volume per feed cycle (depends on which column in series) is regenerated through evaporation and then condensation. Thus, only a small quantity of make-up water is required. Resin replacement is not expected to be a high cost. However, a 20 % per year resin replacement allocation was used in the cost analysis. In contrast, chemical costs are high for precipitation because of acid loss and gypsum landfill disposal.

IE labor cost is relatively low, but power costs are considerably higher than precipitation. IE power costs have been lowered by adopting an economic heat recovery scheme. The IE equipment investment for resin, columns, condensers, and evaporators is the major cost. For the same separation duty, the overall cost of IE is about 40 % less than precipitation.

CONCLUSIONS

Commercially available IR 118 H^+ resin can separate an acid/sugar mixture into its components. IR 122 H^+ resin having twice the crosslinkage but one-half the cost of IR 118 H^+ was also characterized for its partitioning ability. This resin could also be an appropriate candidate for acid/sugar separation.

An existing mathematical model, obtained from literature, was modified and used to simulate IE process acid and sugar elution profiles. Separation performance as a function of process parameters was predicted with this model to find optimum IE processing conditions.

A detailed economic analysis to compare cost for the existing lime precipitation and optimum IE process was performed. The cost comparison analysis strongly indicated that IE can perform the desired separation duty and has several advantages over precipitation. IE not only separates acid from sugar with a 40 % net decrease in overall costs but also eliminates the need of a landfill for waste gypsum. The present studies show that IE is a highly feasible process and strongly suggest that future investigations are justified.

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