Corn Sweetener Refining with Ion Exchange Resins

Ion exchange resins are utilized in the demineralization, enrichment and polishing/decolorization unit operations of high fructose corn syrup refining to produce a pure colorless syrup having the desired sugar profile.

Purolite®

Purolite®

About Purolite

Purolite is a leading manufacturer of ion exchange, catalyst, adsorbent and specialty resins. With global headquarters in the United States, Purolite is the only company that focuses 100% of its resources on the development and production of resin technology.

Responding to our customers' needs, Purolite has the widest variety of products and the industry's largest technical sales force. Globally, we have five strategically located research and development centers and eight application laboratories. Our ISO 9001 certified manufacturing facilities in the United States of America, United Kingdom, Romania and China combined with more than 40 sales offices in 30 countries ensure complete worldwide coverage.

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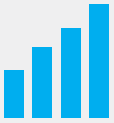
The quality and consistency of our products are fundamental to our performance. Throughout all Purolite plants, production is carefully controlled to ensure that our products meet the most stringent criteria, regardless of where they are produced.

RELIABLE SERVICE

We are technical experts and problem solvers. Reliable and well-trained, we understand the urgency required to keep businesses operating smoothly. Purolite employs the largest technical sales team in the industry.



INNOVATIVE SOLUTIONS

Our continued investment in research and development means we are always perfecting and discovering innovative uses for ion exchange resins and adsorbents. We strive to make the impossible possible.

Corn Sweetener Refining with Ion Exchange Resins

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Demineralization Process in Corn Sweetener Refining

Demineralization of glucose syrup removes ash, protein and color from the solution. It also increases the long-term color stability of the syrup without the need for the addition of sulfur dioxide, which can cause a human allergic reaction in the final consumer products. Ash (calcium in particular) present in the 95% dextrose solution will have a negative effect on the performance of isomerase enzymes and must be removed by demineralization prior to isomerization of a typical 95% dextrose to a 42% fructose solution (42 High Fructose Corn Syrup (HFSC).

Salts are added back into the 95% dextrose solution to facilitate isomerase enzyme performance and are removed after isomerization via demineralization to produce a pure sweetener for sale or for subsequent enrichment to a 55% fructose (55 HFCS) solution.

The ash content of glucose syrups is typically 0.25–0.45% by weight of total syrup dry solids and predominantly contains the following ions:

Sodium Na+, Calcium Ca^{2+}, Magnesium Mg^{2+}, Chloride Cl^{-}, Sulfate SO_4^{2-}

To facilitate isomerization of dextrose to a 42% fructose solution, salts are added back to the syrup after demineralization and will be present in the 42% fructose. These salts must be removed prior to final evaporation or chromatographic separation. The ash content of 42 HFCS is typically 0.15–0.25% by weight of total syrup dry solids and consists primarily of:

Sodium Na+, Magnesium Mg^{2+}, Sulfate SO_4^{2-}, Sulfite SO_3^{-}

As the dextrose or fructose syrup solution passes through the resin bed, the sugars, ash, color bodies and proteins diffuse into the resin beads and can be exchanged or adsorbed onto the resin. In the strong acid cation bed, sodium, calcium, magnesium and other cations will replace the hydrogen ions on the resin due to their greater affinity for the resin than hydrogen ion. The syrup then passes through a bed of weak base anion resin where the mineral acids, organic acids and color bodies diffuse into the resin beads and are adsorbed onto the tertiary amine functional groups.

Strong Acid Cation Service Exchange Reaction

$RSO_3^-H^+ + Na^+ Cl^- \rightarrow RSO_3^-Na^+ + H^+ Cl^-$ **(Produces Mineral Acids)**

Weak Base Anion Service Exchange Reaction

$RCH_2N(CH_3)_2 + H^+ Cl^- \rightarrow RCH_2NH^+Cl^- (CH_3)_2$ **(Acid Adsorber)**

("R" indicates the resin matrix)

The weak base anion resin has a negligible mobile OH^- counter ion which can be exchanged by an anion in solution. Without this ability to split neutral salts, the weak base anion resin must rely on the cation resin to produce acids for it to remove ions by acid adsorption. While the affinity of the cation resin for highly dissociated salts is good, the affinity for proteinaceous materials is much weaker and the cation resin will leak higher percentages of proteins during service.

To prevent proteins and organic acids from affecting the finished product quality, the syrup is typically passed through a secondary cation/anion pair for more complete removal of the impurities leaking from the primary demineralizer pair. Upon reaching the service exhaustion point of the primary demineralizers, the primary pair is regenerated, the secondary pair is moved into primary demineralization service and a third pair of freshly regenerated demineralizers is placed into secondary demineralization service.

Another equipment arrangement for glucose and fructose demineralization is a demineralizer two-bed pair coupled with a polishing strong base anion (Type II) mixed bed for even greater removal of the soluble impurities at the expense of some chemical efficiency. The higher product quality can increase isomerase enzyme life and provide a better feed for the fructose enrichment system. Upon exhaustion, the syrup is displaced from the resin bed and the resin is restored to a working condition with a chemical regeneration treatment. The metal ions are displaced from the cation resin by the passage of a strong mineral acid in an amount in excess of the stoichiometric exchange capacity of the resin in order to drive the equilibrium reaction. Hydrochloric acid is widely preferred to regenerate cation resins by displacing the metal ions and stripping off the proteinaceous compounds. The acids adsorbed onto the weak base anion resin are neutralized utilizing a base such as sodium hydroxide, sodium carbonate or aqueous ammonia. This acid neutralization is accomplished with a smaller excess of regenerant chemical than is required for the cation resin.

Regeneration of the resins is accomplished in a simultaneous sequence in a typical downflow service non-packed bed system as follows:

1. Syrup is displaced from the cation/anion pair by the introduction of condensate or demineralized water into the cation column which then pushes the syrup out through the anion. This step continues until the anion effluent syrup concentration has decreased to 0.1–0.5% dry solids.
2. Process water is passed in an upflow direction simultaneously in both the cation and anion columns to fluidize the resin.
3. A dilute hydrochloric acid solution passes through the bed exchanging hydrogen ions for the metal cations fixed onto the resin.
4. A dilute basic solution passes through the resin bed neutralizing and releasing the adsorbed acids.
5. At intervals of once per 5 to 25 cycles, strip off proteins adsorbed onto the cation resin utilizing caustic or ammonia. Organic acids adsorbed onto the anion resin are stripped utilizing an alkaline brine or hydrochloric acid solution.
6. Water passes through the resins and pushes out the regenerant chemical at a rate that ensures enough contact time for regeneration completion.
7. Condensate or demineralized water is used to rinse out the residual chemical in the beds.
8. Condensate or demineralized water passes through the cation and anion beds in series in a once-though or recirculating manner until the effluent quality reaches the desired conductivity limit of 10–30 microsiemens/cm (µS/cm).
9. Syrup is passed in series through the cation and anion beds displacing the rinse water. This ends when the anion effluent dry solids concentration reaches in excess of 95% of the feed syrup solids concentration.

Strong Acid Cation Regeneration Exchange Reaction

$RSO_3^-Na^+ + H^+ Cl^- \rightarrow RSO_3^-H^+ + Na^+ Cl^-$ (Equilibrium Controlled)

Weak Base Anion Regeneration Exchange Reaction

$RCH_2NH^+ Cl^-(CH_3)_2 + Na^+ OH^- \rightarrow RCH_2N(CH_3)_2 + Na^+ Cl^- + H_20$ (Acid Neutralization)

("R" indicates the resin matrix)

TABLE 1 Demineralizer Sequence

Operation	Solution	Flow Rate (Bed Vols/Hr.)	Volume (Bed Vols)
1. Sweeten Off	H_2O	2.0–4.0	1.5–3.0
2a. Backwash Cation	H_2O	1.0 ft/min	1.5–2.0
2b. Backwash Anion	H_2O	0.33 ft/min	1.0
3a. Chemical in Cation	7% HCl	1.0–1.5	1.0
3b. Chemical in Anion	4% NaOH	1.0–2.0	1.5
4a. Cleanup Cation	4% NaOH	1.0–2.0	1.0
(Alternate)	3% NH_3	1.0–1.5	1.5
4b. Cleanup Anion	7% HCl	1.0–1.5	1.0
(Alternate)	1% NaOH/10% NaCl	1.0	1.5
5a. Slow Rinse Cation	H_2O	1.0–1.5	1.5
5b. Slow Rinse Anion	H_2O	1.0–2.0	1.5
6a. Fast Rinse Cation	H_2O	10–20	2.0–4.0
6b. Fast Rinse Anion	H_2O	10–20	3.0–5.0
7. Series Rinse	H_2O	5–10	2.0–5.0
8. Sweeten On	30–50% DS Syrup	2.0–4.0	1.0
9. Secondary Service	30–50% DS Syrup	2.0–4.0	30–60
10. Primary Service	30–50% DS Syrup	2.0–4.0	30–60

Demineralization Equipment

The demineralization equipment train typically consists of three cation/anion pairs or two cation/anion/mixed bed triplexes. The equipment utilizes food-grade rubber-lined carbon steel pressure vessels containing two or three sets of distributors made of CPVC or stainless steel and wrapped with polypropylene or a stainless-steel screen or Johnson wellscreen. In anion columns, which do not see a hydrochloric acid cleanup solution, stainless-steel distributors are often employed for greater strength against the large shrink/swell and pressure drop forces in the bed. Piping manifolds utilize polypropylene-lined carbon steel, rubber-lined carbon steel or stainless-steel pipe. Control of fluid direction is accomplished utilizing lined and stainless-steel plug valves, butterfly valves or diaphragm valves. Service flow can proceed either down through the resin beds, maintaining them packed in the lower half of the vessels, or up through the resins, maintaining them in a packed state in the upper half of the vessels.

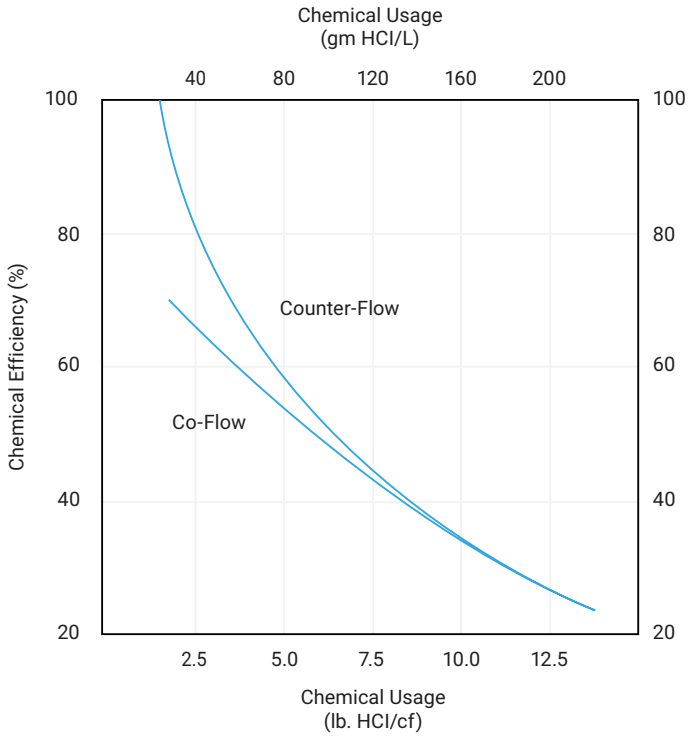
Compartmentalized vessels with external backwash are also utilized. The vessels typically operate with a “water dome” (water fills the freeboard space between the middle and top distributors) to improve rubber lining life and minimize microbiological activity in the vessels during service.

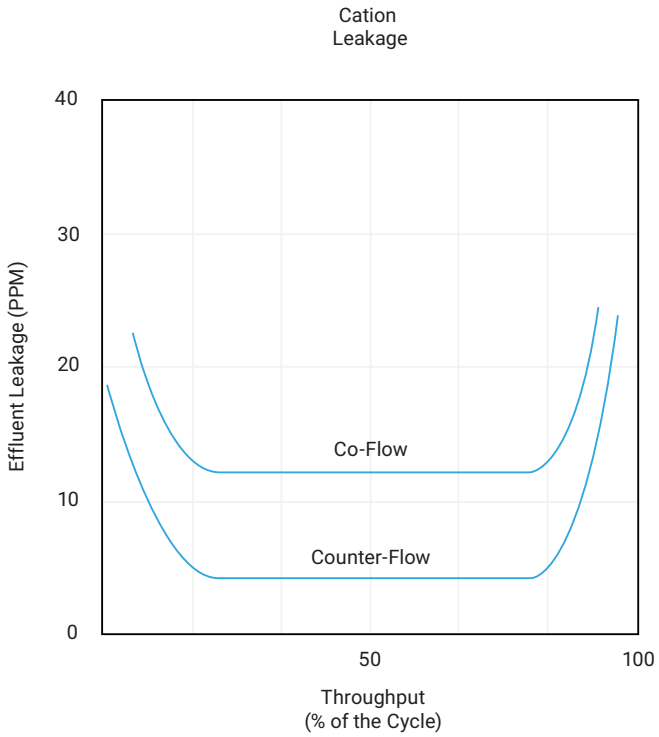
Improved product quality can be obtained from the cation resin by utilizing regenerant chemical passed in a direction counter to the service flow.

Since the syrup leaving the cation column is in equilibrium with the resin located at the outlet collector, the counter-flow passage of regenerant chemical creates an area of highly regenerated resin at the column effluent. At constant chemical dosage, the cation effluent quality is constant and contains fewer impurities than a co-flow regenerated cation resin bed.

FIGURE 1

Cocurrent and Countercurrent Cation HCl Efficiency and Leakage During Service Cycle





If the backwash step can be utilized infrequently, then the resin bed will be undisturbed during regeneration and a counter-flow regeneration may produce a high-quality syrup with the same throughput capacity at a somewhat lower chemical dosage. Since a weak base anion regeneration is an acid neutralization step and only a small excess of regenerant is required, no advantage is gained from counter-flow regeneration of the weak base resin.

Chromatographic Separation Process in Corn Sweetener Refining

What is Chromatographic Separation?

Different sugars passing through a bed of strong acid cation resin in the calcium or sodium form will separate from one another chromatographically due either to a difference in affinity for the resin or to different rates of diffusion into and out of the resin beads. This separation technique can be used to create solutions which have sugar profiles that provide the desired sweetness, taste or physical properties for a consumer product.

A 55% fructose solution will match the sweetness of sucrose when used in soft drinks. This sweetener is produced by passing a 42% fructose solution through a calcium form strong acid cation resin to effect a separation and create a 75–90% fructose solution which can be blended back with additional 42% fructose to produce a 55% fructose purity.

Another class of sweeteners produced utilizing chromatographic separation is the sugar alcohols. Hydrogenation of sugars to produce sugar alcohols such as sorbitol, mannitol, maltitol, erythritol, xylitol or polyols requires high purity feedstocks in order to avoid unwanted byproduct sugar alcohols. As a common example, 95% dextrose is enriched to a 99.4%+ dextrose purity on a sodium or potassium form strong acid cation resin prior to hydrogenation to sorbitol.

Fructose Enrichment

In the production of 55 HFCS from dextrose, an economical limit of 42–46% fructose is achieved using isomerase enzyme. To obtain a higher purity fructose solution, the dextrose and fructose must be separated to produce two fractions, both of which are enriched in either sweetener.

This is accomplished via chromatographic separation on a fractionation ion exchange resin. The fructose-rich fraction can be blended into a 55% fructose solution while the dextrose-rich fraction is recycled in the HFCS refining process.

Owing to a greater number of sites available for hydrogen bonding, the fructose molecule will form a coordination complex with calcium ions fixed onto a strong acid cation resin. This results in a preferential affinity of the resin for the fructose molecule over the glucose molecule and hence a chromatographic separation of the two sweeteners as they pass through the resin bed. From a feed solution containing a purity of 42% fructose by weight, the fructose in the product fraction can reach in excess of 99% purity.

When producing 55% fructose, the optimum productivity and efficiency of the system is achieved by enriching to an 85–90% fructose concentration. Blending this product with a 42% fructose solution to produce the 55% fructose will match the sweetness of sucrose in soft drinks. When enriching to produce crystalline fructose, a product purity in excess of 95% fructose is desired prior to the crystallization step.

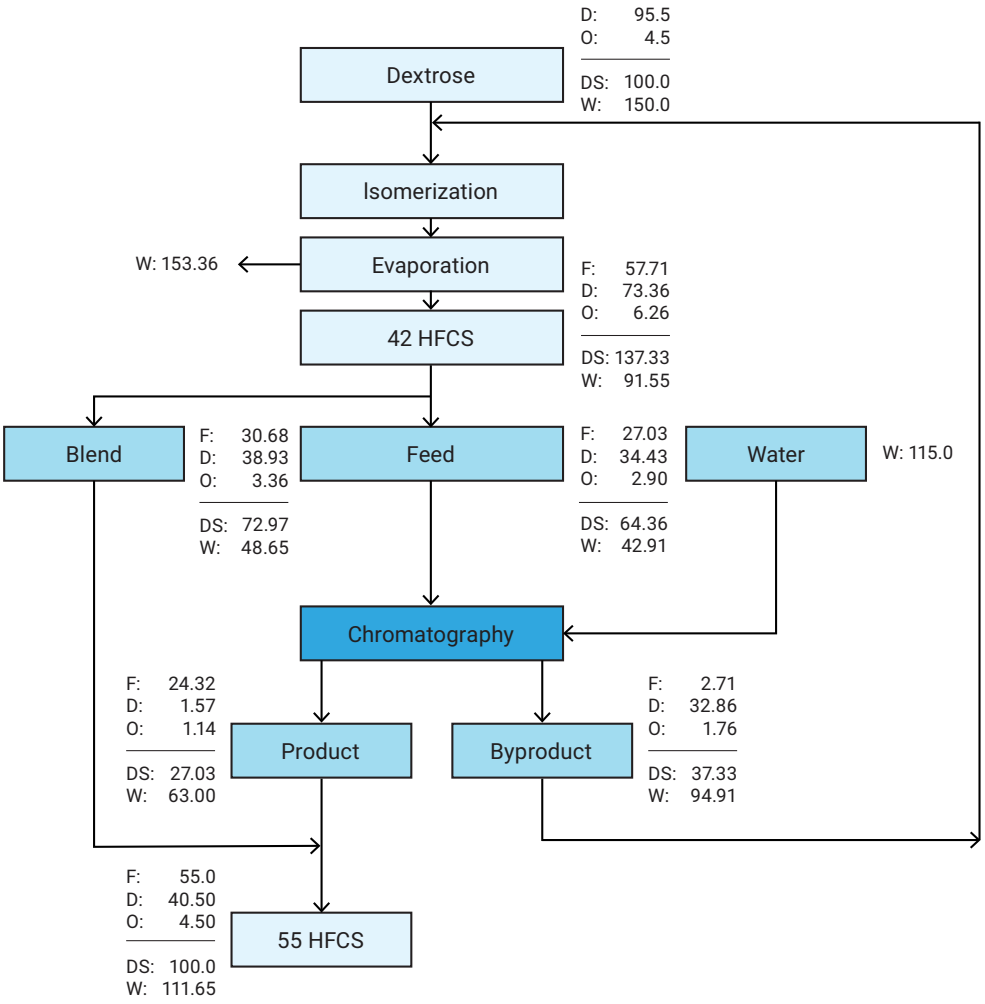
When enriching fructose to produce 55 HFCS with a simulated moving bed chromatographic separation system, a product purity of 90% fructose can be achieved at a 90% recovery of the fructose in the feed steam and a desorbent consumption of 1.1–1.25 lbs. water per lb. of 55 HFCS dry solids.

The production achieved will be approximately 200 lbs. of 55 HFCS dry solids/cu. ft. resin per day depending on the type of system and resin utilized. At constant production and desorbent consumption the purity and recovery will vary inversely with each other.

Chromatographic separation of dextrose is also commercially practiced to separate the dextrose from the oligosaccharides and produce a dextrose purity in excess of 99% in order to minimize unwanted byproducts in the subsequent hydrogenation to sorbitol or in fermentation. Dextrose separation from oligosaccharides occurs due to a difference in the rate of diffusion into and out of the monovalent form cation fractionation resin.

FIGURE 2

5 HFCS Chromatographic Separation Material Balance



F = Fructose
D = Dextrose
O = Oligosaccharides
DS = Dry Solids
W = Water

Chromatographic Separation System

Fructose and dextrose enrichment had initially been accomplished in batch separation systems where the introduction of a pulsed volume of feed was both preceded and followed by recycle fractions and then eluted with desorbent water. In the 1970s, the technology was developed for continuous chromatographic separation of fructose utilizing a simulated moving bed (SMB) separation system.

The SMB technology employs continuous feed and desorbent introduction and continuous product and byproduct withdrawal into and out of a recirculating fluid flow. The recirculating fluid flow can range up to six times greater than the feed flow rate.

In the SMB system, fructose purity in the resin bed upstream from the feed introduction point increases as the distance away from the feed point increases. From a feed inlet reference point, it appears that the adsorbent media is moving upstream and carrying with it the fructose molecules which it has adsorbed, thus the terminology, "simulated moving bed."

The SMB separation system is divided into four process zones with each zone having a unique flow rate. A fifth zone is added when the withdrawal of a polysaccharide rich stream is desired.

Once a stable concentration profile has been established across the entire length of the separation system it moves slowly down the system with the aid of the recirculation flow. The sweetener concentrations are maintained constant by moving the location of the feed, desorbent, product and byproduct inlet and outlet points down the system at the same rate as the concentration profile moves.

Movement of the introduction and withdrawal points is accomplished using either a multiport valve or with multiple manifolds of two-position valves.

Auxiliary systems complement the chromatographic separation system. Degasification of feed and desorbent streams prior to introduction to the resin bed ensures minimal oxidation of the resin will occur. Calcium salt addition to the separator feed is sometimes utilized to maintain a high calcium ion content on the resin, but this increases the ash load to the polishing mixed beds by a small amount.

FIGURE 3

Zones in a Fructose Enrichment SMB Profile

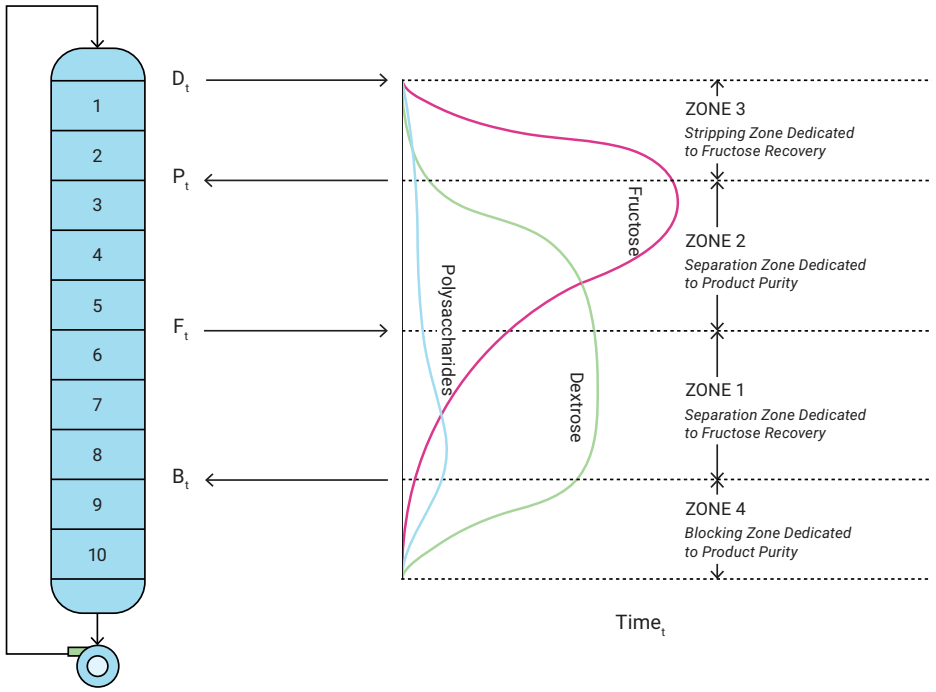
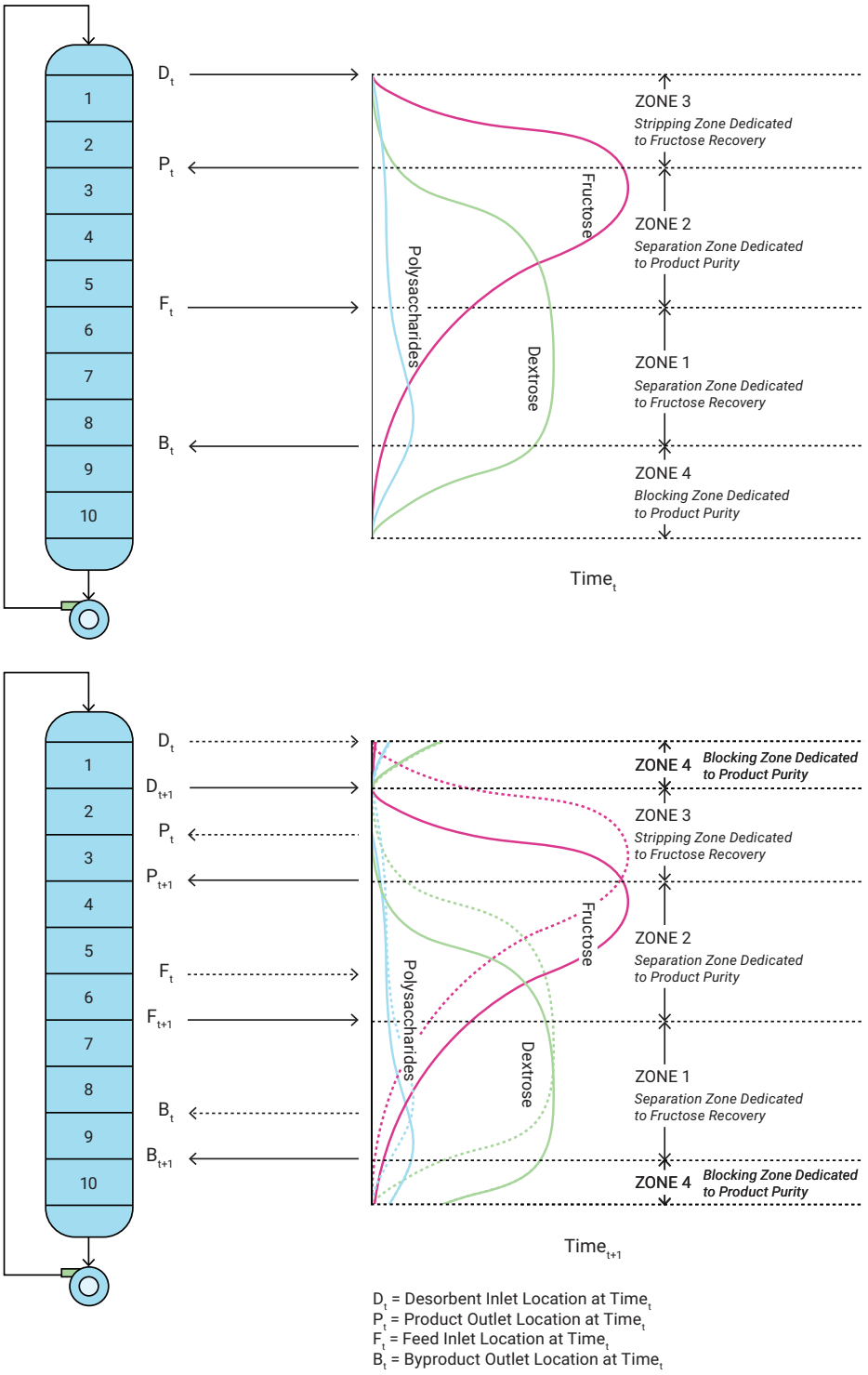


FIGURE 4

Adjustment of Inlet and Outlet Location in an SMB



The Mixed Bed Polishing Process

Color, hydroxymethylfurfural (HMF) and residual ash are removed with ion exchange resins from the fructose-rich product before or after blending to 55 HFCS.

The fractionation system product fraction contains 85–90% fructose and can also contain ppm quantities of impurities such as color bodies, weak acids, HMF (hydroxymethylfurfural), residual ash and protein. Impurity levels can vary because of the fractionation system operating conditions or performance of upstream refining unit operations.

To produce a heat-stable product of consistently high quality, the syrup can be polished by passage through a single bed of strong base anion resin or a mixed bed of strong acid cation/ strong base anion resin. The syrup can be treated as a 90% fructose solution or, after blending, as a 55% fructose solution.

A salt form strong base anion resin will remove color bodies to produce a heat-stable product. These strong base anion polishers can be operated with a single bed in service while the second bed of the pair is in regeneration or on standby. Salt regeneration with occasional caustic cleanup is enough to remove color. For complete removal of weak acids, HMF, ash and protein in addition to color, a mixed bed of hydrogen form strong acid cation resin and hydroxide form strong base anion resin (Type II) is utilized.

In order to more closely match the capacity of the cation and anion resins, a 50% excess of strong base anion resin volume is required to approximately equalize the number of anion exchange groups with the number of cation exchange groups in the mixed bed column since the capacity per unit volume of the strong base anion resin may be 40–50% lower than the cation resin.

Thus, the mixed bed resin volume will typically be 60% anion and 40% cation.

Some degradation of the HFCS can occur in the mixed bed due to contact with the high pH strong base anion resin. Fructose will convert to psichose at elevated pH, so to limit the amount of fructose lost in the mixed beds, the velocity of the syrup is kept high enough to minimize contact time with the anion without affecting the kinetics of the mixed bed exchange.

In service, syrup passes down through one bed of homogeneously mixed cation and anion resin while the second column of the pair is in regeneration or on standby. The primary function of a polisher is color removal, and service is terminated based on color leakage. The conductivity of the mixed bed effluent at breakthrough will be on the order of 1–2 µS/cm. Upon exhaustion of a polisher, the service unit is sweetened off and regenerated while the standby unit is sweetened on and put into service.

To regenerate an exhausted mixed bed unit, syrup is displaced from the resin bed with water, the intermixed resins are separated from each other due to density differences with a backwash and each is then chemically regenerated. The cation resin is stripped of protein and ash with a dilute hydrochloric acid solution while the strong base anion resin is stripped of color bodies, weak acids, ash and HMF with a dilute caustic solution.

Mixed Bed Polishing Regeneration Sequence in Corn Sweetener Refining

Sweeten Off

Syrup is displaced from the bed of intermixed cation/anion resin by introduction of demineralized water into the feed distributor which pushes the syrup down through the bed and out to sweetwater collection. This step is terminated when the effluent sweetener concentration has decreased to 0.1–0.5% dry solids.

Blowdown

The water in the freeboard space between the feed distributor and backwash collector is displaced by pushing the water down through the bed with pressurized air until the liquid level has reached the feed distributor point. Evacuation of the freeboard space results in less back pressure and hence a more rapid rise of the bed of mixed resin during the initial minutes of the backwash step. This provides for a better separation of the more dense cation resin from the lighter anion resin.

Backwash/Separation

Process water is passed upflow through the bed of mixed resins to achieve a 100% fluidized expansion of the resin bed and accomplish a separation of the cation from the anion resin in addition to removing resin fines and particulates from the bed. Owing to specific gravity differences, the anion resin will be fluidized to a greater height in the vessel and settle at a slower velocity than the heavier cation resin. Thus, the cation and anion resin separate into two discrete beds with the resin interface occurring at the same level in the vessel as the interface takeoff distributor.

Chemical-In Anion

The dilute caustic solution continues to enter the feed distributor located above the separated resin bed and passes down through the anion bed and out the interface collector while a simultaneous flow of deionized water enters the bottom distributor and passes up through the cation resin and out through the interface collector with the spent caustic solution. The 50% excess volume of strong base anion resin requires a regenerant chemical volume in excess of the cation regenerant volume. In order to ensure equal contact times per unit volume of resin, regeneration of the anion resin begins prior to the cation resin.

Chemical-In Cation/Anion

The dilute caustic solution continues to enter the feed distributor located above the separated resin bed and passes down through the strong base anion resin and out the interface collector while a simultaneous flow of dilute hydrochloric acid enters the bottom distributor and passes up through the cation bed and out the interface collector with the spent caustic solution.

Slow Rinse Cation/Anion

Demineralized water streams simultaneously enter the feed and bottom distributors and pass down through the anion and up through the cation respectively and mix as they exit the column through the interface collector. The demineralized water displaces the regenerant chemicals from the resins at a rate that ensures that all of the resin receives an adequate regeneration contact time.

Fast Rinse Cation/Anion

Demineralized water streams simultaneously enter the feed and bottom distributors and pass down through the anion bed and up through the cation bed respectively and out the interface collector to rinse out the residual chemicals from the bed. Due to the excess volume of strong base anion resin, and its higher rinse requirement per cubic foot, it is desirable to rinse the anion resin at a higher rate than the cation resin.

Blowdown

Pressurized air enters the top head of the vessel and pushes the water in the freeboard space down through the resin until the liquid level reaches the feed distributor point. Lowering the liquid level prevents water and resin from being carried out the vent nozzle during the subsequent resin mixing steps.

Air/Water Mix

An air/water mixture enters the bottom distributor and flows up through the bed producing a churning action which mixes the resin. The air escapes through a vent while the added water raises the liquid level in the column slowly. The water provides a strong initial hydraulic force to slightly fluidize the resin, so the churning air bubbles will easily affect mixing of the cation and anion resins.

Air Mix

After the churning action is initiated with the air/water combination, the water flow rate is terminated, and the resins continue mixing through the action of the air bubbling up from the bottom distributor and escaping out through the vent.

Air Draindown

In order to settle the well-mixed resins without incurring a separation due to differences in terminal settling velocity, air continues to be introduced into the bottom or interface distributor with the vent valve closed while water is withdrawn from either the bottom or interface distributor.

Fill

Demineralized water enters the top distributor to completely fill the mixed bed vessel with water.

Service Rinse

Demineralized water enters the top distributor and passes down through the freeboard space and through the resin until the effluent conductivity decreases to less than 1 microsiemen/cm. The speed at which the conductivity declines, and the value attained serves as a check against the quality of the regeneration.

Sweeten On

Syrup enters the feed distributor under a demineralized water dome and passes down through the intermixed resin bed and out the bottom collector until the effluent dry solids concentration equals 95% of the feed solids concentration.

TABLE 2 HFCS Mixed Bed Polisher Sequence

Operation	Solution	Flow Rate (Bed Vols/Hr)	Volume (Bed Vols)
1. Sweeten off	H_2O	2.0–4.0	1.5–3.0
2. Blowdown	Air	4	1
3. Backwash	H_2O	0.9 ft/min	2
4. Chemical-In Anion	4% NaOH	2	1
Water-In Cation	H_2O	2	1
5. Chemical-In Cation	7% HCl	2	1.33
Chemical-In Anion	4% NaOH	2	1.33
6. Slow Rinse Cation	H_2O	2	2
Slow Rinse Anion	H_2O	2	2
7. Fast Rinse Cation	H_2O	5	5
Fast Rinse Anion	H_2O	5	5
8. Blowdown	Air	4	1
9. Air/Water Mix	Air	5–6 scfm/sq ft vessel area	10 min
Air/Water Mix	H_2O	0.24 ft/min	–
10. Air Mix	Air	5.5 scfm/sq ft vessel area	10 min
11. Air Drain Down	Air	5.5 scfm/sq ft vessel area	1–5 min
12. Fill	H_2O	2.0–4.0	1
13. Service RInse	H_2O	2.0–4.0	0.5–1.0
14. Sweeten On	30–55% DS Syrup	2.0–4.0	1
15. Service	30–55% DS Syrup	2.0–4.0	35–100

Mixed Bed Polisher Equipment

The mixed bed polishing equipment typically consists of a pair of food-grade, rubber-lined pressure vessels containing four sets of distributors. The distributors are constructed of stainless steel and CPVC piping wrapped with polypropylene screen and clamped to rubber-lined steel support bars. The vessels contain a flat false bottom for resin support and sight windows in the sidewall for visual inspection of backwash expansion, separation and mixing of resins during regeneration.

The piping manifolds utilize polypropylene lined carbon steel, rubber-lined carbon steel or stainless-steel pipe. Control of fluid direction is accomplished with lined and stainless-steel plug valves, diaphragm valves or butterfly valves.

Decolorization, Taste and Odor Removal in Corn Sweetener Refining

Starch hydrolyzate plants around the world are utilizing high surface area synthetic adsorbents to replace granular and powdered activated carbon for removal of color, taste, odor, HMF (hydroxymethylfurfural) and other impurities from sweetener solutions. The Macronet™ line of chemically regenerated styrenic adsorbents offers economic, aesthetic, purity and ease of process advantages for replacing carbon in the production of bottler's quality syrup.

Purolite adsorbents for corn sweetener refining and improving corn sweetener quality include:

- Macronet MN102
- Macronet MN152
- Macronet MN502

Organic impurities are attracted and held to adsorbents by surface energies such as Van der Waals forces. Since adsorption is a surface phenomenon, the Macronet adsorbents are manufactured with large surface area in the range of 800–1100 square meters/gram. But molecules are three dimensional and not flat, so it is important for the surface area to conform to the molecular size of the impurities being adsorbed for the collective surface energy to be large enough to retain them. Thus, the surface area must be employed in micropores which are small enough to form an "adsorption cavity."

Most of the surface area of Macronet adsorbents are contained in adsorption cavities of less than 20 Angstroms diameter. Macronet also contains a significant population of large transport pores which facilitate rapid diffusion from the bulk fluid into the microporous region where adsorption occurs.

The hydrophobic styrene/divinylbenzene matrix of the Macronet will readily adsorb nonpolar hydrophobic impurities. Since many of the impurities are polar or even ionizable molecules, the presence of hydrophilic ion exchange functional groups on the matrix improves the range of impurities which can be removed by attracting more hydrophilic compounds. The hydrophilic functional groups also improve the ease of regeneration of the adsorbent.

Macronet Operating Options

Color Adsorber for Non-Demineralized Syrups

Macronet MN152 replaces conventional carbon powdered treatment of glucose syrups. Expensive and dirty powdered carbon and carbon filters are replaced with a simple chemically regenerated unit operation which produces no solid discharge to handle.

Taste and Odor Polishing

The primary and secondary ion exchange pairs remove the vast majority of impurities, leaving the MN500 available to remove the difficult taste and odor impurities and polish the color even further.

Color, Taste and Odor Polishing

A layered bed of MN152 over MN502 offers improved color and heat color removal in addition to taste and odor polishing.

Color, Taste and Odor Polishing with Enhanced pH and Conductivity Stability

The layered bed of MN152 over MN502 is air mixed prior to service to offer mixed bed quality pH and conductivity.

Color, Taste and Odor Polishing with Enhanced pH Stability

A layered bed of MN152 over a weak acid cation resin is air mixed prior to service to offer better pH stability.

TABLE 3 Service and Regeneration Sequence Macronet MN102, MN152 or MN502

Step	Solution	Temp (°C)	Flow (BV/hr)	Volume (BV)	Time (min)	Comments
Service	Syrup	40–60	2–5	30–200	Variable	Downflow
Sweeten Off	Demineralized water or condensate	40–60	2–5	2	Variable	Downflow
Backwash[1]	Demineralized water or condensate	30–60	2.5–3.7 gpm/ ft^2 of vessel area	1.5–2.0	30	Upflow 50% Expansion
NaOH In[2]	1N NaOH	40–60	1	1.5	90	Downflow
Slow Rinse	Demineralized water or condensate	40–60	2	2	60	Downflow
HCl In[3,4]	0.1N HCl	40–60	2	3	90	Upflow
Slow Rinse	Demineralized water or condensate	40–60	2	2	60	Upflow
Fast Rinse[5]	Demineralized water or condensate	40–60	4	4	60	Downflow
Sweeten On	Syrup	40–60	2–5	1	Variable	Downflow

[1]For MN502, the backwash flow rate should be increased to 6.0 gpm/sq ft
[2]For HMF removal the temperature should be increased to 110 °C and the first 1 BV is allowed to soak for 2 hours.
[3]For MN502 regeneration, the HCl concentration should be increased to 0.3 N.
[4]For a 1/3 MN102 or MN152 and 2/3 MN502 layered bed, the HCl concentration should be increased to 0.2 N.
[5]For mixed bed operation, an air mix follows the fast rinse

Ion Exchange Resin Characteristics

This section covers several important topics regarding the characteristics of ion exchange resin. The topics include a resin specification explanation, resin life expectancy, resin degradation process, resin structure and particle size distribution.

Resin Specification Explanation

Process performance and operating life of resins are optimized through the specification of the resin characteristics which affect them. They cannot, however, be independently optimized. Resin characteristics having a positive effect on operating capacity can have a negative effect on the useful life of the resin. Additionally, a set of characteristics optimized for one particular syrup refining application may not perform effectively for the purification of another type of syrup solution. Optimization of resin characteristics for each type of application is achieved through the specification of resin manufacturing variables. These variables include total capacity, salt splitting capacity, moisture, macroporosity, microporosity, average particle size and particle size uniformity. These characteristics affect resin performance in the following ways:

Total Capacity

Total capacity is a general indicator of operating capacity, but the two are not directly proportional. For a new resin in a syrup demineralization application, the operating capacity is typically 50–60% of the total theoretical capacity. The resins are limited by equilibrium and kinetic constraints from achieving their total theoretical capacity in typical syrup refining operations.

Salt Splitting

Also known as strong base capacity, salt splitting affects the physical stability of weak base anion resins by limiting the amount of reversible swelling which occurs upon exhaustion and also affects the resin's moisture level.

Moisture Level

Moisture levels inside the resin beads affect the rate of diffusion of soluble ions and molecules into the resin bead where exchange or adsorption can occur. Higher moisture content in a resin improves the rate of diffusion.

Macroporosity

Macroporosity also facilitates diffusion into and out of the interior of the resin bead. Large organic molecules have greater difficulty diffusing out of gel resins, thus resulting in a higher degree of fouling. Additionally, macroporosity imparts flexibility to the resin bead which gives it greater physical durability when subjected to osmotic shock or mechanical stresses. Gel resins, not having macroporosity, are more susceptible to osmotic shock and mechanical attrition.

Microporosity

The microporosity of the structure is determined by the degree of crosslinking and affects both ionic and molecular selectivity due to steric hindrance and water adsorption.

Particle Size

Particle size affects the kinetics of ion exchange. Smaller resin beads have shorter film diffusion and particle diffusion path lengths for ions and molecules to travel.

Uniformity of Particle Size

Uniformity affects both pressure drop through the bed and the sharpness of the adsorption and desorption wavefronts. As the particle size distribution widens, the adsorption and desorption bandwidths also increase.

Life Expectancy

The useful operating life of resin is limited by the physical and chemical degradation and fouling which occurs as a result of the particular operating conditions it is subjected to. These conditions include service and regeneration temperatures, syrup dry solids concentration, organic load, cleanup regeneration frequency, type of regenerant chemical and selection of the criteria for the point of replacement.

TABLE 4 Resin Life Expectancy

Resin	Type	Typical Life (No. of Cycles)
A103S, A123S, A133S	WBA	300–400
C150S	SAC-Macro	1000–2000
A510S	SBA-Type II	200–300
PCR642Ca	Fractionation	5–15 years

The Degradation Process and Resin Structure in Corn Sweetener Refining

Successful ion exchange refining of corn sweeteners will result in degradation of the resins in several ways. The predominant mechanism of degradation will differ depending on the type of resin, type of service and choice of regenerant chemical. The table below lists the predominant mechanisms of degradation for each type of resin product offered by Purolite.

TABLE 5 Degradation Mechanisms

Resin	Mechanism	Cause
Cation	Fouling	Irreversible Protein Adsorption
	Oxidation	Dissolved Oxygen in Feed
WB Anion	Fouling	Irreversible Organic Acid Adsorption Cation Oxidation Products
	Thermal	High Operating Temperature Rapid Temperature Change High Rinse Temperature
	Osmotic Shock	Rapid Change of Electrolyte Concentration Rapid Exchange of Ions
	Mechanical Attrition	Resin Abrasion High-Pressure Drop Across Bed
	Physical Loss	Backwash Overfluidization Leaking Distributor Screens
SB Anion – Type II	Thermal	High Operating Temperature High Regeneration Temperature
	Fouling	Irreversible Organic Acid Adsorption
Fractionation	Oxidation	Dissolved Oxygen in Feed or Desorbent
	Mechanical Attrition Osmotic Shock	Resin Abrasion Rapid Change in Syrup Solids Concentration
	Breakup	Re-Wetting Dried Resin with Water Only Rapid Thawing of Frozen Resin

In addition to the chemical mechanisms of degradation, the resin is also subject to breakdown through mechanical attrition and operational problems such as resin retaining screen failures and over fluidization during backwash.

When a resin is in static equilibrium, the chemical bonding forces holding the copolymer network together are balanced by the osmotic swelling pressure from hydration of the functional groups attached to the polymer network. A rapid change in the ionic or syrup concentration of the solution will cause an osmotic pressure that is greater either inside or outside of the bead which cannot be relieved through solvation. This will result in stresses on the polymer backbone that can fracture and break resin beads. A rapid exchange on the resin of one type of ion with another ion of greatly differently hydrated radius will cause the resin to rapidly shrink or swell. These osmotic shock forces occur during each ion exchange or fractionation cycle during sweetening on, sweetening off and regeneration.

Fouling of resins can occur as a result of irreversible adsorption of organic molecules or precipitation of salts within the resin matrix. When large molecular weight organic compounds become sorbed in the resin bead, they can block the pores from further diffusion of ions or bind to an exchange site and prevent utilization of resin capacity. Organic acids with carboxylic groups can become irreversibly sorbed onto the resin due either to a high affinity or to a stereo-chemical effect. The carboxylic group will pick up sodium ions from a caustic regenerant solution which will hydrolyze very slowly off the resin during the fast rinse. This “caustic cling” can result in an extended rinse requirement that is several times higher than new resin rinse requirements.

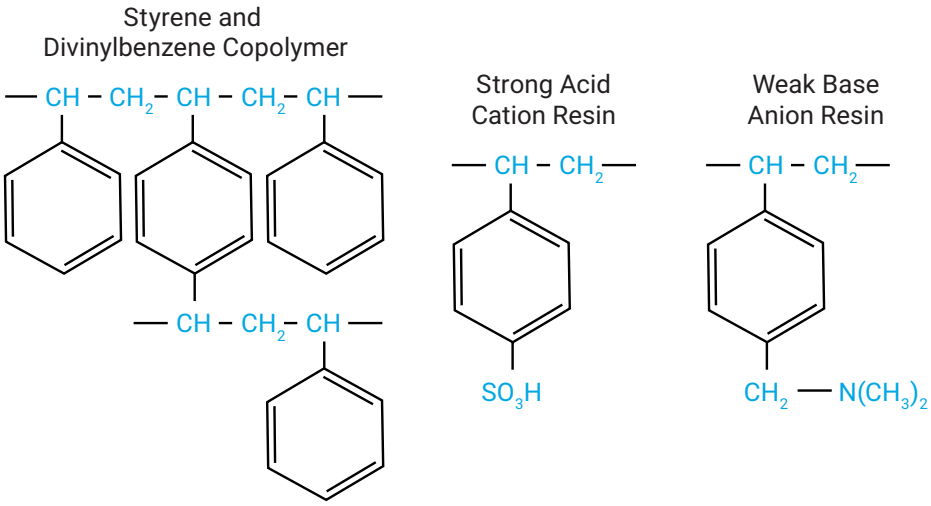
The increase in rinse requirement is a measure of the degree of fouling. Precipitation of magnesium hydroxide can occur within a cation or anion resin bead causing high ion leakage and low capacity. The magnesium hydroxide will precipitate in a cation resin undergoing a cleanup regeneration with caustic if it is not acid stripped first. Magnesium hydroxide precipitation can occur in an anion resin if the rinse water is not softened or if the cation resin is overrun during service.

Resin Structure

The vast majority of ion exchange resins utilized in corn sweetener refining are copolymers of styrene and divinylbenzene which have been activated with sulfuric acid or one of a number of amine compounds to produce cation and anion resins, respectively:

FIGURE 5

Styrene-Divinylbenzene Cation and Anion Resin Structure

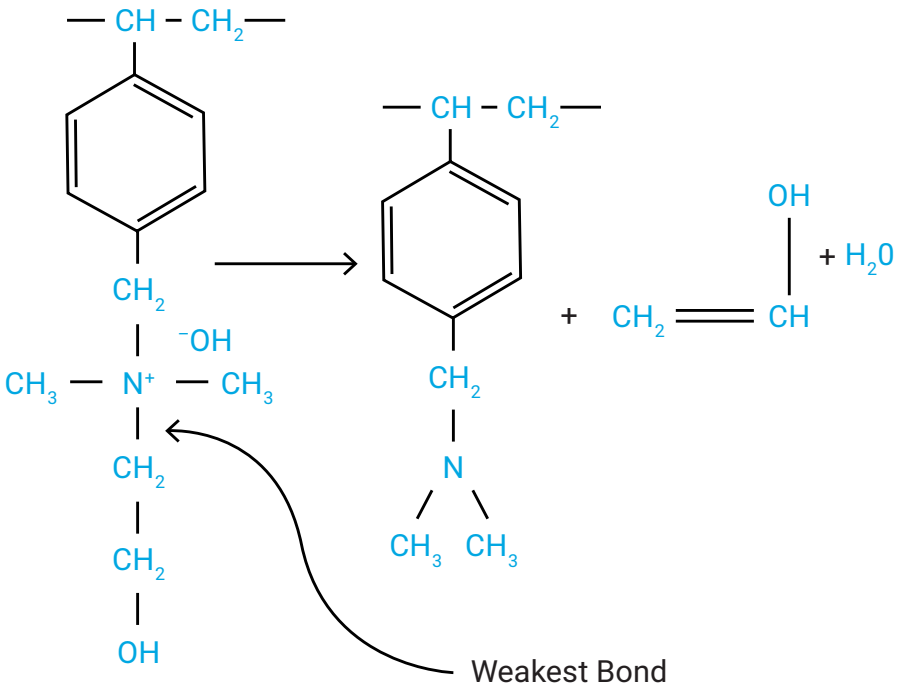


The insoluble resin matrix and bonded functional groups are capable of exchanging ions or adsorbing molecules from a solution and thereby, can affect a change in the ionic or molecular concentrations of the syrup. These properties have made the use of ion exchange resins critical in HFCS refining.

When the temperature of the resin during service or regeneration becomes excessive, the rates of the degradation reactions which result in loss of functional groups increase to a significant level. Product degradation, rather than resin degradation, will limit the cation resin operating temperature, but the strong base anion resin will start to degrade significantly before the syrup. The Type II strong base anion resin in the polishing mixed beds is the least thermally stable resin. The most thermally susceptible bond on a Type II strong base anion resin is the bond holding the alcohol group to the nitrogen atom.

FIGURE 6

Hoffman Degradation of Strong-Base Resins (Type II Strong Base Anion)



Particle Size Distribution of Ion Exchange Resins in Corn Sweetener Refining

Particle-size distribution of ion exchange resins is determined by putting a representative sample through a series of standard sieves. The results are usually expressed as percent of the entire sample which is retained by or allowed to pass through specified openings in the sieves. The most useful data are obtained from resins in their fully swollen states. Wet-screen analyses are generally preferred to and are more consistent than dry-screen analyses. Since the swelling of ion exchange resins can be considerable, any report of particle-size distribution from screen analysis should be accompanied by a statement indicating whether the data were obtained wet or dry.

Particle size and size distribution are sometimes expressed in terms of "effective size" and "uniformity coefficient," both of which may be obtained from screen analyses. Effective size is defined as that opening in millimeters that retains 90% (or passes 10%) of the total resin sample. Uniformity coefficient is the numerical value obtained by dividing the sieve opening (in millimeters) which retains 40% of the sample by that which retains 90%.

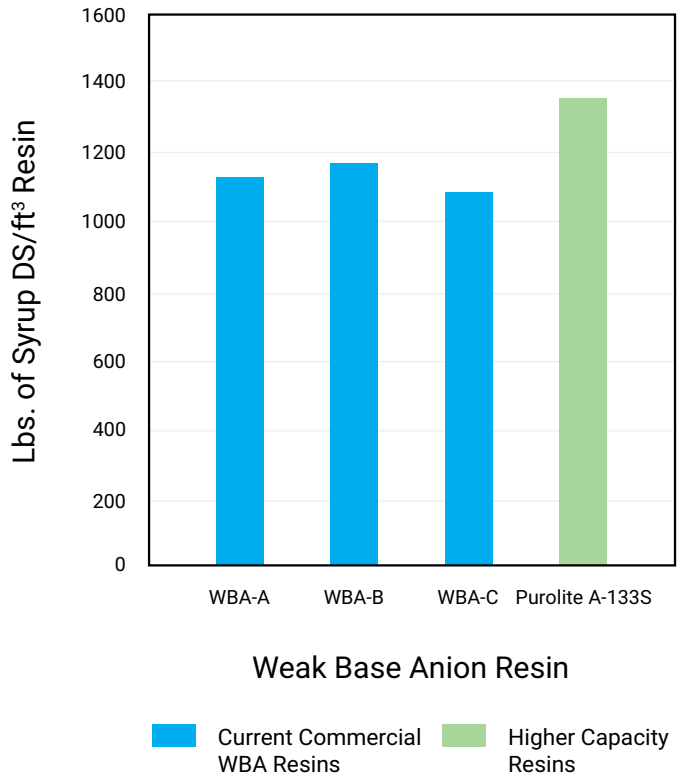
Higher Performance Weak Base Anion Resins

Purolite A133S has been developed to provide 20–25% more throughput per cycle than current commercial weak base anion resins using the same regeneration procedure, resulting in savings that exceed the cost of the resin itself (see figure on the next page).

Purolite A133S gives superior performance in glucose, dextrose, fructose, polyols, maltodextrin and other hydrolyzate syrups

FIGURE 7

High Capacity Weak Based Anion Resin Comparison



Troubleshooting Ion Exchange Resin Performance in Corn Sweetener Refining

While most operational problems with ion exchange systems are the result of mechanical failure or improper conditions of the ion exchange equipment and support systems, there are a number of process problems that can be more difficult to diagnose and correct.

Below is a list of some common ion exchange process related problems, the bolded terms act as the conditions for diagnosing them and the text that follows is the possible causes for those problems. If you have more questions about a product-related issue, please don't hesitate to reach out to the Purolite team.

Low Throughput

Low throughput is caused by any number of failures such as poor fluid distribution or collection, incomplete regeneration, low resin volumes, fouled resins, inaccurate volume totalization or microbiological growth in a resin bed.

High Pressure Drop

High pressure drops are most commonly caused by resin fines not being backwashed out, plugged distributors or strainers or high syrup viscosity due to a drop-in temperature or increase in dry solids concentration. They can also be caused by foreign particulates such as carbon, diatomaceous earth or microbiological growth plugging the bed or laterals.

Excessive Rinse

Organic fouling, as well as poor fluid distribution or collection can be caused by excessive rinse. In addition, high rinse or service temperature may cause thermal degradation.

Resin Breakup and Poor Rinse Water Quality

Excessive service pressure drops across resin bed, as well as osmotic shock from rapid volume change or electrolyte concentration change can cause either of these problems.

High Conductivity or pH in Service

These issues are typically caused by incomplete regeneration, bleed through from leaking service or chemical valve, poor fluid distribution or collection, resin fouling or degradation, excessive service flow rate or inappropriate mixing of cation and anion resins.

Alkaline Degradation of Syrups on Weak Base Anion Resin

Alkaline degradation of syrup can occur as a result of contact with strong base anion groups on the weak base anion resin when regenerated with NaOH. Degradation can be avoided by rinsing regenerated weak base anion resin with dilute NaCl after the slow rinse step, regenerating with soda ash or liquid ammonia or by using a weak base anion resin with no strong base groups.

ICUMSA Method for Color Measurement

This method was originally developed for sugar, but it also used for sweetener measurement. Below, you will find the method for color measurement as used by the International Commission for Uniform Methods of Sugar Analysis (ICUMSA) described in detail. However, it is necessary to know basic information about the equipment used during this process first.

A spectrophotometer can measure the intensity of light over a specific part of the spectrum; usually as transmitted by a substance. It's a relatively simple piece of machinery that utilizes an illumination source, interference filters (powered by a motor), a detector, and a readout device. Note, this type of apparatus won't be necessary for more routine measurements; a photometer using a filter with a narrower bandwidth is suitable. The goal should be to eliminate the inclusion of forward-scattered light in the measurement of transmitted light. By restricting the size of the receiving aperture, only the restricted beam is accommodated.

When measuring white sugar, ICUMSA recommends using cells with a length of ten centimeters. If a test with distilled water shows that two cells are within 0–0.2% of being identical, a second reference cell may be used.

Kieselguhr, a form of diatomaceous earth, should be used as the analytical grade reagent during your procedure.

The sugar to be tested is dissolved in unheated distilled water. The following concentrations are used:

- White Sugars: 50g–100g
- Darker-Colored Sugars: As high as practicable, consistent with reasonable filtration rates and cell depths.
- Liquor, Syrups and Juices: Diluted to 50% solids or original density, unless dilution is required to obtain reasonable filtration rates or cell depths.

The solution is filtered under vacuum; white sugar solution and light-colored liquors are filtered through a membrane filter, pore size 0.45µm. Slower-filtering solutions are filtered with Kieselguhr (1% on solids) through filter paper. The first portion of the filtrate is discarded if cloudy. The pH of darker-colored solutions is adjusted to 7.0±0.2 with dilute hydrochloric acid is removed under vacuum or in an ultrasonic bath, care being taken to minimize evaporation. The density of solution is checked after de-aerating. Distilled water filtered through a membrane filter is used as a reference standard.

The measuring cell is rinsed three times with the sugar solution and then filled. The absorbency of the solution is determined at 420nm using filtered distilled water as the reference standard for zero color. The cell length is chosen so that the instrument reading will be between 0.2 and 0.8 absorbancy, except for solutions of white sugar, where the cell length should be as long as possible.

At the conclusion of your experiment, the molar absorption coefficient A_s is calculated as follows:

$$a_s = \frac{-\text{Log}(T_s)}{(bc)} = \frac{A_s}{(bc)}$$

Where the variables are defined as follows:
T_s = Transmittance
A_s = Absorbance
b = Cell Length in centimeters
c = Concentration of total solids in mols per liter determined refractometrically and calculated from density
a_s = Molar absorption coefficient in liters per mol-cm

Cleaning of Organically Fouled Anion Resins

This process involves a partial caustic regeneration and displacement, followed by a 150 °F, 15% NaCl treatment. The process is repeated until the maximum color eluted during the brine step drops to 1/5 of the highest color eluted during the first treatment. Details of the procedure described above are as follows:

The anion bed is backwashed, then regenerated with 2%–5% NaOH. The amount of caustic must be limited to approximately 1/3 of the normal dosage. The flow rate should be about 0.2 gpm per ft^3. For ten minutes after this step is completed, a slow rinse or displacement should occur at the same 0.2 gpm per ft^3. Then, 150 °F, 10–15% NaCl solution should be injected at 6.5–8 pounds per ft^3. This should also be at a 0.2 gpm per ft^3 flowrate. Finally, the "slow rinse or displace" step described earlier should be repeated. This time, however, note the time at which the eluted color is the highest. This observation should be about the most concentrated salt in the effluent. This process should be repeated (without backwashing) until the color eluted during the salting period is 1/5 of that observed during the first treatment.

It is important to realize the procedure is best completed on a regular or periodic schedule before the anion resin is appreciably fouled. If organic matter in the affluent is high, the above steps should be taken every 15 to 30 days. Note that if the underdrain is made of stainless steel, the HCl will attack it, and the resin will have to be moved to an alternate treatment vessel.

FDA Conditioning of Ion Exchange Resin Before Food Use

Ion exchange resins tend to be insoluble and infusible polymers. Even immediately after processing, however, there are still more soluble impurities which should be removed prior to most applications. The removal can take place by many methods which include a simple water rinse using several bed volumes of water, a chemical regeneration followed by a water rinse, and the most rigorous cleanup which would include an acid/alkali cycling period.

As part of the preparation, Purolite cation resins are steam cleaned to remove all residuals to meet the United States F.D.A. Standards CFR-21 Para.173.25 of the Food Additives Regulations. This procedure is not recommended for anion resins in the free base or hydroxide form. Anions steamed or boiled in the above forms can cause extreme loss of capacity over a very short period.

Anion resins with amine functional groups have a slight odor, especially after storage in highly heated or closed containers. This odor can be washed out and usually only persists through a few cycles.

The standard ionic forms for Purolite resin are sodium form for the strong acid cations, hydrogen form for the weak acid cations, chloride form for the strong base anions and the free base form for the weak base anions. Other ionic forms are supplied by Purolite upon request.

Resins are prepared for conditioning like the ion exchange regeneration procedure:

1. Resins should be transferred to the column and soaked in water for approximately one hour, allowing the resin to come to equilibrium.
2. Backwash the resin to reclassify the bed so that the finer particles are on the top and the coarse particles are on the bottom.
3. Cease backwashing and allow the bed to settle. Then drain the water to 1 inch above the bed.
4. Follow general conditioning steps.

To a bed of resin in the normal backwashed, settled and drained condition:

1. Add three bed volumes of 4% NaOH at a rate sufficient to allow 45 minutes contact time.
2. Rinse with five bed volumes of potable water at the same flow rate.
3. Add three bed volumes of 10% H_2SO_4 or 5% HCl at a flow rate sufficient to allow 45 minutes contact time.
4. Rinse with five bed volumes of potable water.
5. Convert the resin to the ionic form desired for use, using the normal regeneration techniques.

The above conditioning treatment is for all acidic and basic ion exchange resins with the following modifications.

Cation exchange resins to be used in the H^+ cycle are conditioned as outlined. If they are to be used in the Na^+ cycle, the above order of application of acid and base are reversed. If the equipment involved will not tolerate acid, the following substitutions can be made in the conditioning steps as tabulated: In step three, substitute 25 bed volumes of 0.5% $CaCl_2$ for the 10% H_2SO_4 or 5% HCl, or exhaust with tap water. In Step one, substitute 10% NaCl for the 4% NaOH.

Anion exchangers to be used in the chloride or hydroxide cycle can be conditioned as outlined above. Again, it is recommended that chloride conversion using 10% NaCl be used in place of the 10% sulfuric acid in Step 3.

Sanitization of Resins

Peracetic acid, a derivative of hydrogen peroxide, is a good treatment against a wide variety of microbes. Research has shown that peracetic acid will be used to an ever-increasing degree in the field of medicine due to its anti-bacterial, fungicidal, sporicidal and anti-viral action.

Work done by the Degussa Technical Applications Department in conjunction with Chemiewerk Homburg AG determined that peracetic acid is suitable as a disinfectant for deionizers because of its wide spectrum of attack. Using a 0.1% peracetic acid solution in water with a reaction time of one hour, a slime and mold concentration of 104–105/mL was reduced to almost zero. The short rinsing time after using peracetic acid is of importance (typically about 45 minutes or 10–15 BV).

If peracetic acid is used as a disinfectant, the following procedure should be used for both cation and anion resin.

- Ensure anion resins are fully exhausted as peracetic acid performs best at a pH < 8.
- Make up 1 bed volume (BV) of peracetic acid solution containing 0.1% peracetic acid.
- Inject 1 BV of disinfectant at a flow rate of 5 BV/h (0.6 USGM/ft^3) with displacement discharged to a drain approved for chemical waste disposal.
- When all the peracetic acid has been injected, close all valves and retain the disinfectant for at least one hour to soak the resin and pipe work.
- Carry out a displacement rinse using raw water for at least 60 minutes at 5 BV/h, followed by a fast flush for 30 minutes.
- Regenerate the resin once and return the unit to service.

Determination of Acidity in Corn Sweetener Refining

Mineral Acidity: pH of 4.3 and below (Steps 1–5)

Total Acidity: Initial pH to pH 8.3 (Steps 1–7)

1. 58.3 ml or aliquot sample.
2. Place in 125 ml white evaporating dish.
3. Add two to three drops of methyl orange.
4. Stirring gently, titrate with .02N NaOH (sodium hydroxide) from red to orange yellow (pH 4.3).
5. The calculation you should now perform is: mineral acid (M.A.) gpg as:

$$CaCO_3 = \frac{\text{ml titre x 58.3}}{\text{ml of sample}}$$

6. To the above sample, add two to three drops of phenolphthalein and continue titration with .02N NaOH until sample becomes pink.
7. The calculation you should now perform is: total acid (T) gpg as:

$$CaCO_3 = \frac{\text{ml titre x 58.3}}{\text{ml of sample}}$$

Note: This test is usually applied to the cation effluent water.

Appendix

For conversions, please reference our Unit Conversion Tool at: https://bit.ly/39fEYaN.

TABLE A Conductance vs. Total Dissolved Solids

% By Weight	ppm	NaCl	NaOH	H_2SO_4	Sea Salt	HNO_3	HCl	HF	Acetic Acid	CO_2	NH_3	H_3PO_4	SO_2
0.0001	1.0	2.2	6.2	8.8	2.2	6.8	11.7	–	4.2	1.2	6.6	–	–
0.0003	3.0	6.5	18.4	26.1	6.5	20	35.0	–	7.4	1.9	14	–	–
0.001	10.0	21.4	61.1	85.6	21.3	67	116	–	15.5	3.9	27	–	–
0.003	30.0	64	182	251	64	199	340	290	30.6	6.8	49	–	–
0.01	100	210	603	805	208	657	1140	630	63	12	84	342	–
0.03	300	617	1780	2180	612	1950	3360	1490	114	20	150	890	–
0.1	1,000	1990	5820	6350	1930	6380	0.0111	2420	209	39	275	2250	3600
0.3	3,000	5690	0.0169	0.0158	5550	0.0189	0.0322	5100	368	55	465	4820	7900
1.0	10,000	0.0176	0.0532	0.0485	0.0170	0.0600	0.103	0.0117	640	–	810	0.0105	0.0172
3.0	30,000	0.0486	0.144	0.141	0.0462	0.172	0.283	0.0347	1120	–	1110	0.0230	0.0327
10.0	100,000	0.140	0.358	0.427	–	0.498	0.709	0.118	1730	–	1120	0.0607	0.0610
30.0	300,000	–	0.292	0.822	–	0.861	0.732	0.390	1620	–	210	0.182	–

Conductivity of various compounds at 25 °C (Microsiemens/cm and Siemens/cm)
Underlined figure indicates conductivity passes through a maximum between the two listed concentrations.

TABLE B Conversions: Capacity and Regeneration Level Equivalents

mEq/ml	Pound Equiv./ft³	Kgr (as $CaCO_3$)/ft³	Grams CaO/l	Grams $CaCO_3$/l
1.00	0.0624	21.8	28.00	50.00
16.00	1.00	349.00	449.00	801.00
0.0459	0.000286	1.00	1.28	2.29
0.0357	0.00223	0.779	1.00	1.79
0.02	0.00125	436.00	0.56	1.00

TABLE C Conversions: Pressure Equivalents

lb/in²	Feet of Water	Meters of Water	Inches of Mercury	Atmospheres	kg/cm²
1.000	2.31	0.704	2.04	0.0681	0.0703
0.433	1.00	0.305	0.822	0.0295	0.0305
1.421	3.28	1.00	2.89	0.0967	0.10
0.491	1.134	0.346	1.00	0.0334	0.0345
14.70	33.93	10.34	29.92	1.00	1.033
14.22	32.80	10.00	28.96	0.968	1.00

TABLE D Conversions: Flow Rate Equivalents

US gpm	ft³/h	m³/h	ft³/s	l/s
1.00	8.021	0.2271	0.0023	0.0631
0.0125	1.00	0.0283	0.0167	0.4721
4.403	35.30	1.00	2118.00	16.67
438.60	60.00	1.70	1.00	28.33
15.85	127.16	3.60	2.12	1.00

TABLE E Physical Constants of 55% High Fructose Corn Syrup

% DS	At 20% Refractive Index	Density (g/ml)	Total Pounds Per Gallon	Total Pounds Solids Per Gallon
3	1.3373	1.0118	8.419	0.253
4	1.3387	1.0157	8.452	0.338
5	1.3402	1.0197	8.485	0.424
6	1.3417	1.0237	8.519	0.511
7	1.3432	1.0277	8.552	0.599
8	1.3447	1.0318	8.585	0.687
9	1.3462	1.0359	8.620	0.776
10	1.3477	1.0400	8.654	0.865
11	1.3492	1.0442	8.689	0.956
12	1.3508	1.0483	8.724	1.047
13	1.3523	1.0525	8.759	1.139
14	1.3539	1.0568	8.794	1.231
15	1.3555	1.0611	8.830	1.324
16	1.3570	1.0654	8.866	1.418
17	1.3586	1.0697	8.902	1.513
18	1.3603	1.0741	8.938	1.609
19	1.3619	1.0785	8.975	1.705
20	1.3635	1.0829	9.011	1.802
21	1.3652	1.0873	9.048	1.900
22	1.3668	1.0918	9.086	1.999
23	1.3685	1.0963	9.123	2.098
24	1.3702	1.1009	9.161	2.199
25	1.3719	1.1055	9.199	2.300
26	1.3736	1.1101	9.238	2.402
27	1.3753	1.1147	9.276	2.503
28	1.3770	1.1196	9.315	2.608

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% DS	At 20% Refractive Index	Density (g/ml)	Total Pounds Per Gallon	Total Pounds Solids Per Gallon
29	1.3788	1.1241	9.354	2.713
30	1.3805	1.1288	9.394	2.815
31	1.3823	1.1336	9.433	2.924
32	1.3841	1.1384	9.473	3.031
33	1.3859	1.1432	9.513	3.139
34	1.3877	1.1481	9.554	3.248
35	1.3895	1.1530	9.593	3.358
36	1.3913	1.1579	9.636	3.469
37	1.3932	1.1628	9.677	3.580
38	1.3951	1.1679	9.718	3.693
39	1.3969	1.1729	9.760	3.806
40	1.3988	1.1779	9.802	3.921
41	1.4007	1.1830	9.844	4.036
42	1.4026	1.1881	9.887	4.152
43	1.4045	1.1932	9.930	4.270
44	1.4065	1.1984	9.973	4.388
45	1.4084	1.2036	10.016	4.507
46	1.4104	1.2089	10.060	4.627
47	1.4124	1.2141	10.104	4.749
48	1.4144	1.2194	10.148	4.871
49	1.4164	1.2248	10.192	4.994
50	1.4184	1.2302	10.237	5.118
51	1.4205	1.2356	10.282	5.244
52	1.4225	1.2410	10.327	5.370
53	1.4246	1.2465	10.373	5.497
54	1.4267	1.2520	10.418	5.626

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% DS	At 20% Refractive Index	Density (g/ml)	Total Pounds Per Gallon	Total Pounds Solids Per Gallon
55	1.4287	1.2575	10.464	5.755
56	1.4309	1.2631	10.511	5.886
57	1.4330	1.2687	10.557	6.018
58	1.4351	1.2743	10.604	6.150
59	1.4373	1.2800	10.651	6.284
60	1.4394	1.2857	10.699	6.419
61	1.4416	1.2914	10.747	6.555
62	1.4439	1.2972	10.795	6.693
63	1.4460	1.3030	10.843	6.831
64	1.4483	1.3088	10.891	6.970
65	1.4505	1.3147	10.940	7.111
66	1.4528	1.3206	10.989	7.253
67	1.4550	1.3265	11.039	7.396
68	1.4573	1.3325	11.088	7.540
69	1.4596	1.3385	11.138	7.685
70	1.4620	1.3445	11.188	7.832
71	1.4643	1.3506	11.239	7.980
72	1.4667	1.3567	11.290	8.129
73	1.4690	1.3628	11.341	8.279
74	1.4714	1.3690	11.392	8.430
75	1.4738	1.3752	11.444	8.583
76	1.4762	1.3814	11.496	8.737
77	1.4787	1.3877	11.548	8.892
78	1.4811	1.3940	11.600	9.048
79	1.4836	1.4004	11.653	9.206
80	1.4861	1.4067	11.706	9.365

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% DS	At 20% Refractive Index	Density (g/ml)	Total Pounds Per Gallon	Total Pounds Solids Per Gallon
81	1.4886	1.4131	11.760	9.525
82	1.4911	1.4196	11.813	9.687
83	1.4937	1.4261	11.867	9.850
84	1.4962	1.4326	11.921	10.014
85	1.4988	1.4391	11.976	10.183

TABLE F Concentration of Regenerants: Hydrochloric Acid

% HCl	Grams Solids/Liter	Normality (eq/l)	Density (g/ml)	° Baumé	Pounds HCl Per US Gallon
1	10.03	0.275	1.0032	0.5	0.084
2	20.16	0.553	1.0082	1.2	0.168
4	40.72	1.12	1.0181	2.6	0.340
6	61.67	1.69	1.0279	3.9	0.515
7	72.30	1.98	1.0345	4.6	0.603
8	83.01	2.28	1.0376	5.3	0.693
10	104.70	2.87	1.0474	6.6	0.874
12	126.90	3.48	1.0574	7.9	1.059
16	172.40	4.73	1.0776	10.4	1.439
20	219.60	6.02	1.0980	12.9	1.833
30	344.80	9.46	1.1492	18.8	2.877
34	397.60	10.90	1.1693	21.0	3.318
40	479.20	13.10	1.1980	24.0	3.999

TABLE G Concentration of Regenerants: Sulfuric Acid

% H_2SO_4	Grams Solids/Liter	Normality (eq/l)	Density (g/ml)	° Baumé	Pounds Solids Per US Gallon
1	10.05	0.205	1.0051	0.7	0.084
2	20.24	0.413	1.0118	1.7	0.169
3	30.55	0.625	1.0184	2.6	0.255
4	41.00	0.836	1.0250	3.5	0.342
5	51.59	1.05	1.0317	4.5	0.431
6	62.31	1.27	1.0385	5.4	0.520
8	84.18	1.72	1.0522	7.2	0.703
10	106.6	2.17	1.0661	9.0	0.890
12	129.6	2.64	1.0802	10.8	1.082
15	165.3	3.37	1.1020	13.4	1.379
20	227.9	4.65	1.1394	17.7	1.902
50	697.6	14.2	1.3951	41.1	5.821
96	1762.0	35.9	1.8356	66.0	14.710
100	1831.0	37.3	1.8305	65.8	15.280

TABLE H Concentration of Regenerants: Sodium Hydroxide

% NaOH	Grams Solids/Liter	Normality (eq/l)	Density (g/ml)	° Baumé	Pounds Solids Per US Gallon
1	10.10	0.262	1.0095	1.4	0.084
2	20.41	0.511	1.0207	2.9	0.170
3	30.95	0.774	1.0318	4.5	0.258
4	41.71	1.04	1.0428	6.0	0.348
5	52.69	1.32	1.0538	7.4	0.440
6	63.89	1.60	1.0648	8.8	0.533
8	86.95	2.17	1.0869	11.6	0.726
10	110.9	2.77	1.1089	14.2	0.925
12	135.7	3.39	1.1309	16.8	1.333
16	188.0	4.70	1.1751	21.6	1.569
20	243.8	6.10	1.2191	26.1	2.035
40	571.9	14.29	1.4300	43.6	4.773
50	762.7	19.10	1.5253	49.9	6.365

TABLE I Concentration of Regenerants: Ammonia

% NH_3	Grams Solids/Liter	Normality (eq/l)	Density (g/ml)	° Baumé	Pounds Solids Per US Gallon
1	9.939	0.584	0.9939	10.9	0.0829
2	19.79	1.162	0.9895	11.5	0.1652
3	29.60	1.741	0.9852	11.6	0.2470
4	39.24	2.304	0.9811	11.7	0.3275
6	58.38	3.428	0.9730	13.9	0.4872
8	77.21	4.536	0.9651	15.1	0.6443
10	95.75	5.622	0.9575	16.2	0.7991
12	114.0	6.694	0.9501	17.3	0.9515
14	132.0	7.751	0.9430	18.5	1.102
16	149.8	8.796	0.9362	19.5	1.250
18	167.3	9.824	0.9295	20.6	1.396
20	184.6	10.84	0.9229	21.7	1.540
22	201.6	11.84	0.9164	22.8	1.682
24	218.4	12.82	0.9101	23.8	1.823
26	235.0	13.80	0.9040	24.9	1.961
28	251.4	14.76	0.8980	25.9	2.098
30	267.6	15.71	0.8920	27.0	2.233

TABLE J Concentration of Regenerants: Sodium Carbonate

% Na_2CO_3	Grams Solids/Liter	Normality (eq/l)	Density (g/ml)	° Baumé	Pounds Solids Per US Gallon
1	10.09	0.1904	1.0086	1.2	0.0842
2	20.38	0.3845	1.0190	2.7	0.1701
4	47.59	0.8979	1.0398	5.6	0.3471
6	63.64	1.201	1.0606	8.3	0.5311
8	86.53	1.633	1.0816	10.9	0.7221
10	111.3	2.081	1.1029	13.5	0.9204
12	134.9	2.545	1.1244	16.0	1.126
14	160.5	3.028	1.1463	18.5	1.339

TABLE K Specifications for HFCS-55

Parameter	Value
Percent Solids	76.5–77.5 g/100 g with a target value of 77.0 g/100 g
Fructose	55.0–58.0 g/100 g of total solids
Dextrose + Fructose	Not less than 95.0 g/100 g of total solids
Other Saccharides (D.P. 2+)	Not more than 5.0 g/100 g of total solids
Ash	Not more than 0.05 g/100 g, sulfated
Taste	Free from foreign taste.
Titratable Acidity	Not more than 4.0 ml of 0.05N NaOH to raise 100 g to pH 6.0
Temperature	Not more than 30 °C at time of receipt
Copper	Not more than 1.5 mg/kg
Iron	Not more than 3 mg/kg
Lead	Not more than 1 mg/kg
Total Heavy Metals	Not more than 5 mg/kg (as lead)
Chlorides	Not more than 50 mg/kg (as NaCl)
pH	4.0 target (undiluted); range: 4.0 ± 0.5
Sulfur Dioxide	Not more than 6 mg/kg
Sulfonated Polystyrene	Not more than 1.0 mg/kg, or must pass test for sulfonates
Odor After Acidification	Free from objectionable odor when one liter of 54° Brix syrup is acidified to pH 1.5 with phosphoric acid (H_3PO_4). Solution is warmed to 30 °C and checked for odor every 5 minutes for 30 minutes duration.
Sediment	Not more than 2 mg/kg visual insolubles when 300 g of solids are filtered through a 28-mm (1–1/8 inch) Whatman No. 54 filter disc and compared to a standard NSDA disc; or not more than 7 mg/kg water insolubles determined by a gravimetric method.
Floc	Shall not produce a floc when one 1 liter of 54° Brix solution, acidified to pH 1.5 with phosphoric acid, is allowed to stand 10 days at room temperature. The solution should be viewed with the aid of a strong beam of light.
Color	Not more than 35 Reference Basis Units (RBU), at time of receipt, as determined in accordance with the NSDA standard for "Bottlers" sugar. A supplier's product must not increase in color to more than 50 RBU after storage for 30 days at 30 °C.
Turbidity	A 54° Brix syrup acidified to pH 1.5 with phosphoric acid shall be free of turbidity when viewed in a 1 liter container under a strong beam of light.
Microbiological	Not more than 5 viable yeast per 10 mL Not more than 5 viable mold (mycelium) per 10 mL Not more than 25 viable mesophyllic bacteria per 10mL By "Direct Method" the count shall not exceed a total number of viable and non-viable organisms as noted: Not more than 10 yeast per 1.0 ml; not more than 10 mold (mycelium) per 1.0 ml; not more than 100 mesophilic bacteria per 1.0 ml

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