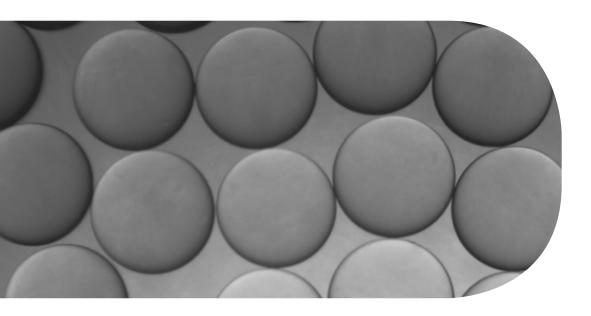


Nutrition
Sweeteners,
Amino Acids and
Organic Acids.



ION EXCHANGE RESINS AND ADSORBENTS IN SWEETENER AND ACIDS PROCESSING

Sugar and starch derived sweeteners are some of the most popular nutrients and can be found in about 80% of our food products. The production and consumption of carbohydrates and sweeteners is steadily increasing. White sugar is probably one of the most popular sweetener and is used in many different aliments. Drivers for this development are the increasing global income, the demand for soft drinks and sweets, the production of bio ethanol and a growing demand for fermentation products as renewable building blocks for the chemical industry.

Carbohydrates can be recovered from all kinds of sources such as sugar beets, sugar cane, corn, wheat, sorghum, tapioca, cassava, cellulose and many other natural sources. Sucrose, glucose, fructose and other polyols are widely used as sweeteners in the food industry. But carbohydrates can also be fermented into organic acids such as citric acid, lactic acid and succinic acid. Ethanol and different building blocks for the chemical industry such as 1,4 butanediol and 1,3 propanediol are produced nowadays to replace petrochemicals and gasoline.

Adsorbents, ion exchange resins and chromatographic resins are widely used in the nutrition, beverage, chemicals and sweetener industry. They allow the removal of colour and production by-products by adsorption, the demineralisation of sugar juices by ion exchange, the recovery of sugars from non-sugars and the separation of different kind of polyols by chromatographic processes.

CHEMRA has developed and is marketing different kinds of ion exchange resins, chromatographic media and adsorbents. They allow economical processes by reducing at the same time the CO_2 footprint of companies. CHEMRA supports efforts to reduce the environmental impact of production processes by supporting high performance processes through tailor made products and new developments. CHEMRA is able to adjust their separation media to allow the most economical process.

CHEMRA consults companies to improve their processes and to reduce their environmental impact.



Organic acid recovery with chromatographic process



STARCH BASED SWEETENERS

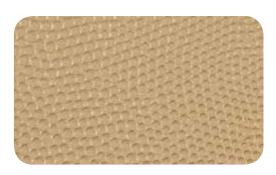
DECOLOURISATION, ION EXCHANGE, MOLASSES DESUGARISATION AND INVERSION

Starch is one of the major sources of solid and liquid sweeteners and polyols. Raw materials are in general corn, wheat, rice, tapioca, rye, barley, cassava and potatoes. Starch is generally hydrolysed by enzymes and the conversion level is adjusted to the requirement of the final product. The syrup is generally de-ashed and decolourised. TREVERLITE resins are used to refine syrups. Proteins and other by-products such as HMF can be removed at the same time, too.

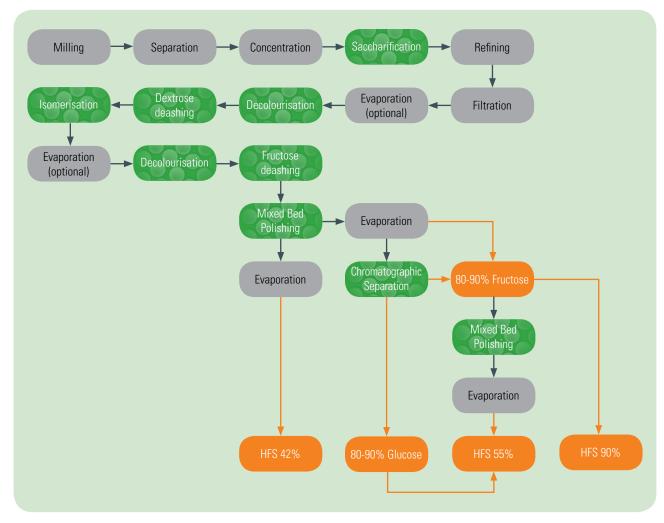
The most widely used source of starch is corn. Starch based sweeteners are primarily high fructose syrups (HFS) which are widely used in canned fruits, soft drinks, cereals, ice cream, dairy products, in drugs, chemicals and pharmaceuticals but also in the production of bio ethanol and organic acids.

The figure to the right shows the production process of starch based sweeteners including the usage of ion exchange resins, enzyme carriers and adsorbents in the production of glucose and fructose. For an overview of the different applications and the suitable ion exchange resins please refer to the table on page 7.

After processing the crop to a slurry, the starch solution is purified and hydrolysed by either enzymes which can be immobilized on a specific carrier or by an acid. Depending on the enzymatic system and process used DE-28 up to DE-98 can be produced. High DE syrups are intermediates for fructose syrups and polyols such as sorbitol but find their major uses in beverages and all kind of foodstuff respectively.



Chromatographic resin for glucose/fructose separation or ion exclusion

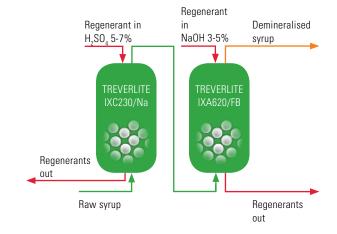


Production process of starch based sweeteners

Adsorbents like TREVERSORB ADS630 and TREVERLITE IXA310/CL are used to remove colour bodies, HMF, Maillard reaction products, soluble proteins and fatty acids. Furthermore undesired off flavours and colour precursors can be removed. In some cases, traces of proteins have to be removed from high fructose syrups to improve the stability of the syrup.

TREVERSORB ADS600 is a cationic adsorbent resin, has a specific porosity and a high surface area to ensure an excellent protein uptake and efficient regeneration.

Cation and anion exchange resins like TREVERLITE IXC230/NA and TREVERLITE IXA630/FB are used to demineralise the solution before further processing. TREVERLITE ion exchange resins are available in different particle sizes for different processes in the upflow and downflow mode.



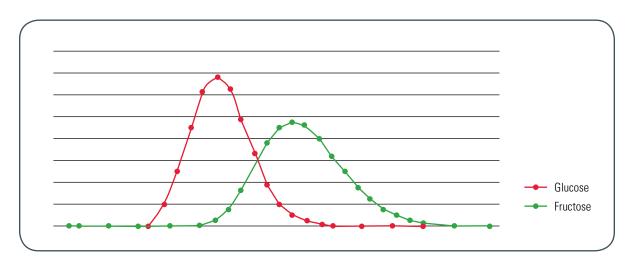
Demineralisation of syrups

High fructose starch based syrups are produced from refined high DE glucose syrups. An isomerase fixed on a TREVERZYME resin carrier enables the conversion of glucose to fructose. Multi column processes are in use in the industry. The isomerase catalyses the conversion of glucose to 42% fructose. This syrup is then often concentrated and used as a general sweetener in many applications.

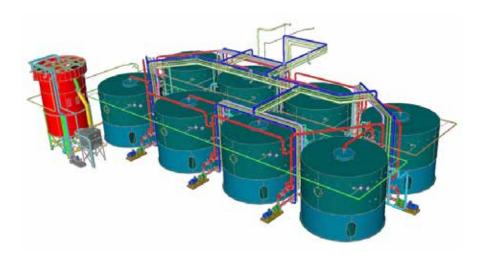
In order to increase the sweetening power of HFS-42 syrup and to match the property of sucrose, the fructose content has to be increased to 55%. By using a chromatographic system preferably with high performance chromatographic

resins such as TREVERCHROM CHR250-PS/CA or TREVERCHROM CHR310-PS/CA, the mixture of glucose and fructose can be separated into two fractions which are rich in either glucose or fructose. The fructose rich fraction is blended back with HFS-42 to obtain HFS-55 or can be concentrated to HFS-90 for low calorie foodstuff or it can be further processed to pure fructose.

The figure below shows the separation of glucose and fructose with TREVERCHROM CHR310-PS/CA.



Separation of glucose and fructose



Recovery of sucrose & betaine from sugar beet molasses



REFINING OF POLYOLS

Sugar alcohols such as sorbitol can be obtained by hydrogenating a clear dextrose solution with a Ni catalyst. This solution is then subsequently purified by ion exchange resins such as TREVERLITE IXC230/NA and TREVERLITE IXA620/FB

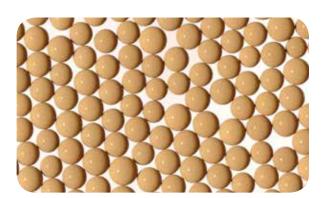
and specific adsorbents or activated carbon. This syrup is then spray dried or crystallised to obtain a powder. Sorbitol has a variety of applications including the manufacture of Vitamin C, the addition to low calorie food and in oral care.

Process	Resin	Properties
Decolourisation	TREVERLITE IXA310/CL	Macroporous styrenic anion exchange resin with good color removal and good regenerability
	TREVERLITE IXA510/CL	Macroporous acrylic anion exchange resin with very good colour removal properties and long life time
	TREVERLITE IXA110/CL	Gel type styrenic anion exchange for high colour uptake in polishing units
Polishing	TREVERLITE IXC230/H/MB	Mixed bed for demineralization
	TREVERLITE IXA620/FB/MB	Consisting of a high capacity macroporous cationic resin and a styrenic weak base anionic resin
Glucose, fructose and polyols deashing	TREVERLITE IXC230/H	High capacity strongly acidic cation exchanger for long cycles
	TREVERLITE IXA620/FB	Styrenic weak base anion for low conductivity products
	TREVERSORB ADS600	Strongly acidic adsorbent for removal of traces of colour and proteins
Protein adsorption	TREVERSORB ADS600	Strongly acidic adsorbent
Inversion	TREVERLYST CAT210	High activity and long life time
Colour and HMF removal	TREVERSORB ADS630	Weakly basic and removes colour and HMF
Saccharification	TREVERZYME	



CANE SUGAR REFINING

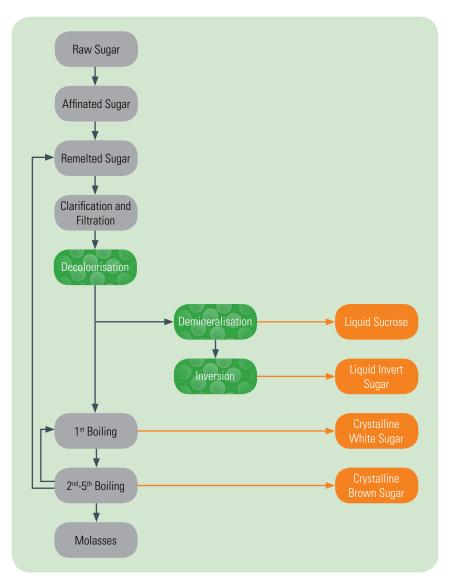
Sucrose is primarily extracted from sugar beet and sugar cane but many different crops contain at least small amounts of sucrose, too. Once the sugar solution is extracted, it still contains minerals and colour bodies which have to be removed through various process steps to reach its universally popular white granulated form. During processing the colour of the sugar solution is increasing due to Maillard reactions, oxidation, enzymatic processes, caramelisation and degradation of sugars and has to be removed.



Macroporous strong acid cation exchange resins for demineralisation

DECOLOURISATION, ION EXCHANGE AND CATALYSIS

Sucrose is extracted from the cane sugar stalk in a cane sugar mill by crushing, squeezing and hot water extraction. The juice contains about 17° Brix, is then purified with lime, flocculants and $\rm CO_2$ and filtered through filter presses or vacuum filters. After concentration to 60-70° Brix the sugar is undergoing the first crystallization ending up with tan coloured raw sugar crystals. This sugar is generally sent to a sugar refinery for further clarification and decolourisation. These refineries are sometimes linked to the mill but are often located close to the consumer markets, too.



Refining process of raw sugar from sugar cane

Sugar decolourisation

Decolourisation of sugar juices can be done through activated carbon or with polymeric ion exchange resins. These ion exchange resins have either an acrylic or a styrenic backbone depending on the inlet colour and the expected degree of decolourisation.

Acrylic anionic resins such as TREVERLITE IXA510/CL have the advantage of allowing a very high load of colour bodies. Due to their hydrophilic backbone they are less sensitive to fouling and can be regenerated very efficiently compared to styrenic resins. On the other hand their selectivity towards the colour bodies is less compared to styrenic anionic resins like TREVERLITE IXA310/CL. Depending on the decolourisation process used and the inlet colour TREVERLITE IRA510/CL is removing up to 60-80% of the colour bodies. Styrenic adsorbents are more selective to colour bodies and can remove up to 70-90%. Sugar solutions of up to 2000 ICUMSA units can be treated economically, but generally the sugar syrups have less than 1000 IU.

Due to their selectivity to the colour bodies styrenic resins are more sensitive to fouling when overloaded with colour and may not be used when the sugar solutions contain more than 800 ICUMSA units. A shorter lifetime is the result of overloading the resin.

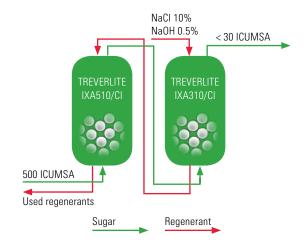
Both, TREVERLITE IXA310/CL and TREVERLITE IXA510/CL have a specific macroporous structure allowing excellent colour uptake and a very efficient regeneration ¹⁾. In some cases gel type acrylic strong base resins such as TREVERLITE IXA410/CL can be used to achieve a higher degree of decolourisation compared to porous resins.

Colour bodies are captured primarily by hydrophobic interactions of the colour molecule with the aromatic or aliphatic structure of the resin and by van-der-Waals forces.

lon exchange and the formation of hydrogen bonds are responsible to a lesser degree.

Many plants are using a combination of acrylic and styrenic resins utilising them both at their optimum. By combining the properties of both resins, they are gaining the advantage of a longer resin life and highest decolourisation efficiency. In some cases when very low colour is required a polishing mixed bed resin bed with a strong base gel type anion such as TREVERLITE IXA110/CL in combination with a weak acid cation such as TREVERLITE IXC310/H is required. This anionic resin has a high decolourisation performance but is rather sensitive to fouling, the weak acid resin is adjusting the pH and is catching some additional impurities. Colour levels of down to 20-30 IU can be achieved. The mixed bed is reducing the ash content at the same time to the required level.

The regeneration is generally performed at a slightly alkaline pH with a 10% NaCl / 0.1-0.5% NaOH solution. The salt is pushing out the colour bodies from the pores and surface of the resin partially by dewatering the colour molecule and is breaking the van-der-Waals forces between resin surface and colour molecule. The slightly alkaline pH is allowing an ion exchange reaction to take place and prevents the degradation of sucrose.



Cane sugar decolourisation

¹⁾ For more information about highly efficient colour removal plants with a very low CO₂ footprint please consult your local CHEMRA technical expert.

Case study "Cane Sugar Decolourisation"

The data of this case study are based on the long-term performance of a commercial cane sugar decolourisation unit. The incoming sugar has a colour load of 800-1,000 IU 1). The sugar solution can be decolourised by about 80% in the first step through the acrylic resin TREVERLITE IXA510/CL down to 200 IU. The second step can further reduce the colour to less than 50 IU. A major focus in this plant was given to the reduction of waste waters and energy by reducing the resin inventory and resin consumption at the same time. It can be seen that the consumables and waste streams have been significantly minimized. The amount of waste water even though being already low can be further reduced by using a combination of a RO and NF. A zero discharge operation with minimal product losses and energy consumption has been developed getting to a very low CO₂ footprint.



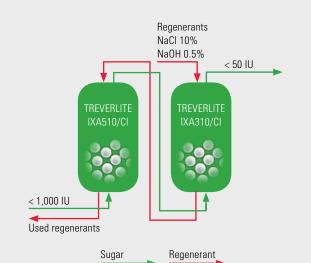
Brine recovery with nanofiltration

Colour reduction

- $< 1000 \text{ IU}^{-1} \text{ to} < 50 \text{ IU}$
- 5 BV/h
- Colour loading TREVERLITE IXA510/CL ± 50 000 dS colour/m³ resin/cycle TREVERLITE IXA310/CL ± 20 000 dS colour/m³ resin/cycle

Consumables per 100 t of sugar treated 2)

- NaCl 70 kg
- NaOH 10 kg
- HCI 6 kg
- Condensate 10,0 m^{3 3)}
 Mixed waste water 6,0 m^{3 3)}
 Dilute sweet waters 3,5 m^{3 3)}
 Waste water after NF 0,55 m^{3 4)}



 $^{^{1)}}$ IU = measured at 420 nm

²⁾ Lower inlet colour reduces the consumables significantly

³⁾ The effluents can be further reduced with a RO/NF system.

⁴⁾ The overall waste can be further minimised. Depending on the refinery set up a 'zero' discharge process can be provided.

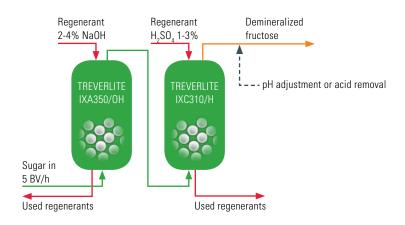
¹⁾ IU = measured at 420 nm

Sugar Demineralisation

Demineralization of sucrose can be done in two different ways. If inversion has to be avoided, a combination of a weak acid ion exchanger like TREVERLITE IXC310/H and a strong base anion exchanger such as TREVERLITE IXA350/OH can be used. They can be used either in a mixed bed operation or separately where the strong base anion exchanger is in the lead position followed by the weak acid cation exchanger. In this case the pH is around 4 and has to be adjusted to avoid inversion. This can be achieved by:

- either blending the solution with a side steam,
- treating the solution with a weak base anionic adsorbent to remove acidity and colour,
- treating the sugar solution with a strongly acidic resin in the Na form or
- by adding diluted NaOH if the ash content allows.

At temperatures below 10°C, a strongly acidic resin may be used followed by a weak base anionic resin. Many combinations of resins are used in this kind of operation.



Deashing of sucrose

Sugar Inversion

Sucrose can be inverted to glucose and fructose by either using a mineral acid or by using a strongly acidic catalyst in the H+ form such as TREVERLYST CAT210. In order to reduce the formation of HMF, a degradation product of fructose which is subsequently creating undesired colour in the sugar solution, the temperature should not be higher than 40°C and the flow rate should be adjusted accordingly. If the ash content is high, it might be advisable to use a mixed bed consisting of a strong acid cation exchanger (TREVERLITE IXC230/H/MB) and a weak base anion exchanger (TREVERLITE IXA630/FB) or a combination of a strong base anion (TREVERLITE IXA350/OH) and a weak acid cation (TREVERLITE IXC310/H). If needed HMF can be removed with a specific adsorbent such as TREVERLITE ADS630.

Brine Recovery

Besides highly sophisticated ion exchange and decolourisation systems which are based on an improved fractal technology, CHEMRA can offer highly performing nanofiltration systems through selected business partners to reduce the environmental impact of decolourisation units. Up to 90% of the brine can be recovered with this process step. By further concentrating the remaining 10% of waste water a "green" plant with almost zero discharge and a very low ${\rm CO_2}$ footprint can be obtained. Depending on the plant configuration zero discharge can be even achieved.

Process	Resin	Properties
Decolourisation	TREVERLITE IXA310/CL	Macroporous styrenic strong base anion exchange resin with good colour removal and good regenerability
	TREVERLITE IXA510/CL	Macroporous acrylic strong base anion exchange resin with very good colour removal properties and long life time
	TREVERLITE IXA110/CL or TREVERLITE IXA210/CL	Gel type styrenic strong base anion exchange for high colour uptake in polishing units
Demineralisation	TREVERLITE IXC310/H/MB TREVERLITE IXA350/OH/MB	Weakly acidic acrylic resin Macroporous strong base anion resin Mixed bed for demineralization at about neutral pH
	TREVERLITE IXC230/H	High capacity macroporous strongly acidic cation exchanger for long cycles
	TREVERLITE IXA620/FB	Weakly basic styrenic anionic resin for low conductivity products
	TREVERLITE IXC310/H	Weakly acidic acrylic resin for pH adjustment and removal of traces of cations
Inversion	TREVERLYST CAT210	Strongly acidic cationic catalyst with porous adsorbent
Colour and HMF removal	TREVERSORB ADS630	Weakly basic porous adsorbent for the removal of colour and HMF
Glucose Isomerases	TREVERZYME	Enzyme carrier for isomerases



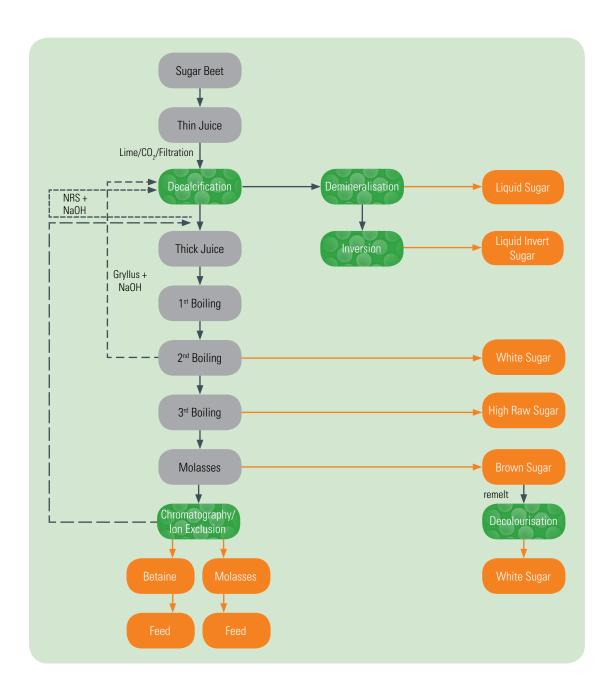
BEET SUGAR REFINING

ION EXCHANGE, DECOLOURISATION, CATALYSIS AND CHROMATOGRAPHY

Sugar beets are one of the major sources of sucrose (saccharose) in Europe. The sugar juice is extracted by hot water from sugar beet slices. This sugar juice contains sugar and non sugars like salts, betaine and colour which will end up in the molasses after crystallisation. Ion exchange resins are widely used in the sugar beet process.

Two major applications can be found in beet sugar refining:

- Decalcification or softening of the thin juice before crystallisation (weak acid cation, NRS and Gryllus process) and
- Recovery of sugar from molasses by a chromatographic process.
 Sugar, glucose and fructose are separated from non sugars using ion exclusion as a separation principle (MDC process). Betaine can be recovered by this process.



Decalcification of thin juice

Decalcification of the thin juice is needed to reduce scaling of the evaporators by Ca/Mg carbonate, oxalate or silicate. The resins of choice are TREVERLITE IXC110/NA and TREVERLITE IXC230/NA.

Three major processes are used to remove hardness from thin juice prior evaporation. The most common technology is right now probably the NRS process where the strongly acidic ion exchange resin is regenerated with soft thin juice. The resin of choice is TREVERLITE IXC110/NA.

The second process installed was in the past the Gryllus process. It is using thick juice after the 1st or 2nd crystallisation to regenerate a high capacity strongly acidic marcropo-

rous resin such as TREVERLITE IXC230/NA. Both processes have the advantage that no additional waste is generated. The used regenerants are either sent back to the carbonatation stage or to the 3rd crystallisation.

More recently a third process gains significant market share. It uses an optimised fractal shallow bed system in combination with a weak acid resin such as TREVERLITE IXC310/H to remove Ca from the thin juice. This process allows a high flow rate at reduced operating cost and minimum product losses. Apparently this process has significant economical advantages compared to the older processes.

Desugarisation of Molasses

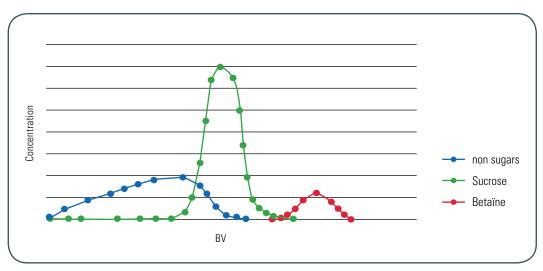
Molasses still contain a significant amount of sucrose, some invert sugar and non-sugars such as betaine and amino acids. Overall this sugar fraction counts for 1/3-1/6 of the refined sugar.

Chromatographic strongly acid resins are used in the K form to separate sugars from non sugars and betaine as additional value added product. The resin of choice is TREVERCHROM CHR310-PS/K. More recently chromatographic resins with a smaller particle size such as

TREVERCHROM CHR250-PS/K or TREVERCHROM CHR280-PS/K have been developed to allow processes with improved flow characteristics and a higher resolution. Fractal based distribution systems can overcome the problem of increased pressure drop due to smaller particle size.

A more efficient separation of the molecules and subsequently a smaller footprint for the plant and less resin inventory are the result of this new development.

Fractionation of beet molasses



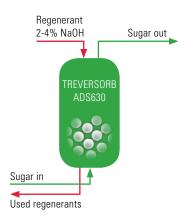
Process	Resin	Properties
Thin juice	TREVERLITE IXC110/NA	Reduction of evaporator downtime
	TREVERLTIE IXC230/NA	Macroporous strong acid cation exchanger with very
		high capacity and improved osmotic stability
NRS Process	TREVERLITE IXC110/NA	Gel type strong acid cation exchanger with good lifetime
Gryllus process	TREVERLITE IXC230/NA	Macroporous strong acid cation exchanger with very
		high capacity and improved osmotic stability
Decalcification	TREVERLITE IXC310/H	Weakly acidic acrylic resin for efficient regeneration
Desugarisation/Ion Exclusion	TREVERCHROM CHR310-PS/K	Gel type chromatographic resin for standard operating systems
	TREVERCHROM CHR250-PS/K and	Gel type chromatographic resins for high end chroma-
	TREVERCHROM CHR280-PS/K	tographic systems



REMOVAL OF COLOUR AND HMF BY SYNTHETIC ADSORBENT

HMF is formed during different steps when processing sugar and fructose solutions at elevated temperatures for an extended time. It is a degradation product of hydrocarbons. Often colour bodies are formed at the same time and have to be removed, too. CHEMRA has developed a specific adsorbent called TREVERSORB ADS630 for the removal of HMF and small amounts of colour bodies. In some processes HMF can be even recovered by using TREVERLITE X910810.





HMF and colour removal

Process	Resin	Properties
Colour and HMF removal	TREVERSORB ADS630	Weakly basic anionic adsorbent for the removal of colour and HMF
HMF recovery	TREVERLITE XS910810	Highly porous adsorbent for the removal and recovery of small molecules

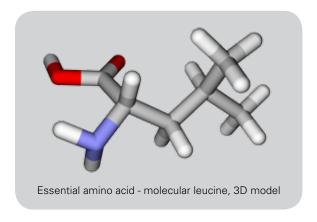
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AMINO ACIDS

Amino acids can be recovered and purified by using ion exchange resins and adsorbents. Typically, lysine is recovered and purified by a high capacity cation exchange resin in the $\mathrm{NH_4}$ form such as TREVERLITE IXC120/NH4.

Other amino acids like methionine, leucine, isoleucione and valine can be recovered and separated by using newly developed chromatographic resins. The CHEMRA technical representatives will provide you with professional advice.







ORGANIC ACIDS

ION EXCHANGE, CATALYSIS, ADSORPTION AND CHROMATOGRAPHY

Industrial organic acids are mostly produced through fermentation processes. The major organic acids separated and purified through ion exchange and chromatographic processes are citric acid, succinic acid and maleic acid.

Traditionally citric acid was purified through cationic and anionic resins but more recently a chromatographic process using medium basic acrylic resins such a TREVERCHROM CHR400-AC/FB has been used.

Bio-based succinic acid can be recovered by using a chromatographic resin such as TREVERCHROM CHR280-PS/H. In case the ester has to be formed to finally produce THF a catalyst like TREVERLYST CAT500 can be used. For the separation, purification and esterification of organic acids from fermentation broths please consult our technical staff.

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Carboxylic acid Alcohol Ester



Separation of an organic acid with a chromatographic process



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For the address of your nearest local sales office please visit our website.

Governmental regulations vary from country to country. Please seek advice from your local representative in order to determine the best resin choice and operating conditions. Food grade resins often require specific and extended certifications.

Operating conditions refer to the use of the product under normal operating conditions. They are based on experience in industrial applications. However, additional data are needed to calculate the resin volumes for ion exchange units. For more questions please contact our technical experts.

Ion exchange polymers and adsorbents are generally of industrial grade and impure except otherwise stated by CHEMRATM. Chemicals and gases must be handled with care and by trained personal, regulatory requirements and safety standards must be met. Oxidative chemicals like nitric acid or peroxides can be explosive in combination with ion exchange polymers and adsorbents, others can be corrosive. Rewetted dry polymers develop heat and expand significantly. CHEMRA makes no warranties either expressed or implied as to the accuracy or appropriateness of this information and technical advice — whether given verbal, in writing or by way of trials — is given in good faith and expressively excludes any liability upon CHEMRA arising out of its use. Our recommendations cannot be seen as recommending the use of the product in violation of any patent or license. We recommend that the prospective users determine for themselves the suitability of CHEMRA materials and suggestions for any use prior to their adoption. Specifications might be subject to change without further notice. Materials safety data sheets and handling methods are available on request.

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