

Chromatographic purification of raw beet juice: A lime-free technology*

Chromatographische Saftreinigung von Rohsaft (aus Rüben) ohne Kalk

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A process for raw beet/cane juice purification by chromatographic treatment of juice was proposed and patented by Amalgamated Research Inc. several years ago. In the beet application the process involves removal of suspended solids from the raw juice stream with further softening, evaporation and chromatographic purification of the resulting syrup. Compared to conventional purification the chromatographic process provides significantly higher nonsugar and color elimination without any use of lime. Using fractal fluid distribution devices has significantly enhanced efficiency of the proposed process by allowing for significant inventory reduction in resin-based separation processes. The possibility of crystallization of high quality white sugar has been demonstrated in several independent experiments. The process also provides overall sugar extraction yield exceeding 92%.

Key words: Juice purification, chromatography

Vor einigen Jahren wurde von Amalgamated Research Inc., USA, ein chromatographisches Saftreinigungsverfahren für Rüben- und Rohr-Rohsäfte vorgeschlagen und patentiert. Für die Rübenzuckerindustrie schließt das Verfahren die Abtrennung der suspendierten Feststoffe aus dem Rohsaft gefolgt von Enthärtung, Eindampfung und chromatographischer Reinigung des Sirupe ein. Verglichen mit der konventionellen Saftreinigung erlaubt das chromatographische Verfahren, bedeutend größere Mengen an Nichtzuckern und Farbstoffen ohne den Einsatz von Kalk zu entfernen. Fraktale Flüssigkeitsverteilungen verbessern den Wirkungsgrad des Verfahrens signifikant, da die Stoffmengen in den Ionenaustauscherkolonnen wesentlich reduziert werden können. Die Kristallisation qualitativ hochwertiger Weißzucker wurde in mehreren unabhängigen Untersuchungen nachgewiesen. Die Zuckerausbeute bei diesem Verfahren liegt bei über 92 %.

Stichwörter: Saftreinigung, Chromatographie

1 Introduction

Several efforts have been made worldwide to replace the lime-based juice purification process adopted by most sugar factories. Although not highly efficient in terms of nonsugar removal (only about 30% nonsugar elimination from the feed stream), the conventional process has yet to be challenged by a more modern technology. New separation methods applied to the sugar process affect so many aspects of traditional sugar production that they must be thoroughly studied to assure that no significant changes in final product quality will be observed. Obtaining reliable information concerning the technical and economical feasibility of proposed processes via long-term pilot testing becomes a very important issue. The latter is complicated by the fact that the quality of beet raw juice changes throughout the processing campaign. The ultimate difficulty for any new technology involving more than one step in the process is finding a way to implement the technology and minimize the risks associated with integration of the new process into existing operations.

Several years ago Amalgamated Research Inc. (ARi) developed and patented a new lime-free raw juice purification technology based on large-scale industrial chromatography (Kearney et al., 1995). The technical feasibility of the new process has been demonstrated with continuous pilot work throughout several campaigns. Results have been reported elsewhere (Kearney and Rea-*rick*, 1996). Several steps in the new process have required signifi-

cant effort and extensive testing to assure scalability of the proposed technologies. Another goal of these studies was to obtain sufficient quantities of associated waste products and evaluate their utilization or disposal opportunities. The latter work was performed within the framework of an international consortium of eight sugar companies. The details of this work are proprietary to the group of sponsors. However, general information required to commercialize the new technology will be discussed in this paper. This will also update the sugar community on the progress of lime-free raw juice purification technology and discuss ways to integrate it into existing production facilities.

2 Process description

2.1 Unit operations

The proposed raw juice purification process includes several unit operations. A block diagram of the process is presented in Figure 1. Raw juice pH value is adjusted to stabilize sucrose. Juice is also heated to initiate the clarification process. Estimates show that it is efficient to remove suspended solids by simple clarification. The underflow stream (mud) is additionally handled to reduce sugar losses. Clarifier overflow containing only a fraction of extra

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fine material is sent to membrane filtration. Depending on membrane selection, some screening device may be needed to protect the membrane channels from plugging. Juice is filtered and sterilized simultaneously by passing through micro- or ultrafiltration membranes. At the same time, large molecular mass components are removed from the juice. Depending on the configuration of the membrane system and the ratio of permeate to retentate, a certain amount of retentate must be treated to minimize the sugar losses. The experiments have demonstrated that overall sugar losses in clarifier mud and membrane retentate can be reduced to a level comparable with lime cake losses in a conventional factory. The raw juice permeate is softened to eliminate divalent cations that may reduce efficiency of the chromatographic resin. The softened juice is evaporated to bring the dry substance content to about 70%. At this stage evaporation is quite efficient; no scale control is required. Highly efficient plate evaporators can be used reliably. The concentrated raw juice is then subjected to chromatographic purification. The high purity stream, extract, is crystallized to yield white sugar. Low extract color (typically below 1000 ICUMSA Units) allows crystallization of white sugar in two consecutive stages. The sugar-lean fraction from the chromatographic purification, raffinate, is evaporated and can be used as a cattle feed. Material balance calculations based on the results of large-scale pilot studies show that the expected overall process extraction exceeds 92% for a raw juice purity of about 88–89%.

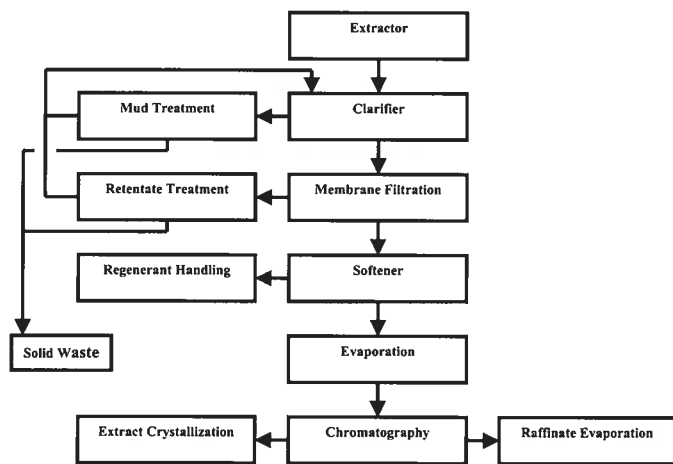


Fig. 1: Process flow diagram

For several campaigns the proposed process was studied on a smaller pilot scale. At that time pilot installation operated continuously at 500 mL/min 24 hours per day seven days a week throughout campaign. Test results were reported in a publication by Kearney and Rearick (1996).

In order to reliably scale-up the proposed unit operations as well as obtain sufficient amounts of waste materials to evaluate potential applications and/or means of disposal it was required to increase the size of the pilot plant. A larger pilot installation was constructed at the Amalgamated Sugar Co. LLC in Twin Falls, Idaho. The pilot simulated all steps of the proposed process. A slip stream of raw juice was fed into a pilot clarifier at 22 m³/h. Several membrane systems, softening and evaporation pilot systems were installed. The size and parameters of each system were carefully selected to assure full scalability to commercial installations. Peripheral equipment, such as screens, heat exchangers, and hydrocyclones were evaluated in the course of the study.

Table 1: Estimate of sucrose balance discrepancy if suspended solids are neglected

	Based on dissolved solids	Corrected for suspended solids
Raw juice purity in %	88	88
RDS (as measured) in %	16	16
Raw juice flow in sht/d	8640.0	8596.8
Suspended solids flow		
at 0.5% concentration in sht/d	.	43.2
Sucrose in raw juice in sht/d	1216.51	1210.43
Difference in sucrose in sht/d	.	-6.08
Difference in \$/campaign*	.	486,400

*160 days campaign, \$500/sht sugar

2.2 Suspended solids removal from raw juice

Key requirement for any resin based separation technology, such as softening or chromatography is removal of all suspended solids from feed solutions. In a conventional process the level of suspended solids in the raw juice stream is not monitored. It is commonly believed that the refractometric method of dry substance content measurement (RDS) is not affected by the presence of suspended solids. Experiments with raw juice streams indicate that an error may be introduced into calculations due to the presence of suspended solids (Kochergin et al., 2001).

Data in Table 1 demonstrate that neglecting suspended solids in the raw juice stream results in a significant discrepancy in the sucrose balance. Since the RDS measurement does not account for suspended solids, they should be subtracted from the total juice flow. Otherwise, the sucrose content in the raw juice will be overstated. For the proposed raw juice purification process, the use of RDS measurements in the material balance calculations would not account for the suspended solids stream. Finding an economic way to remove suspended solids from raw juice was challenging project. Seemingly simple, the task calls for new processes different from conventional filtration. In preliminary tests it was found that use of conventional pressure filtration required about 0.3–0.4% of filter-aid based on juice volume. Estimated cost of required filter-aid and the difficulty of disposal make the process non-feasible. The concentration of suspended solids in the raw beet juice, which depends on the extraction stage, may range within 0.5–1.0% wt. An extremely broad particle size distribution is a characteristic of suspended solids in raw juice.

A clarification method has been developed and patented by ARI (Kochergin et al., 2000) which allows removal of a large portion of suspended solids. The method was validated for several years using a 4 m-diameter clarifier at a feed flow rate of 22.5 m³/h. Figure 2 illustrates the clarifier performance throughout one campaign. Note that the volumetric concentration of suspended solids was monitored on an hourly basis. The solids were routinely measured on a volume basis, because of the difficulty of a mass procedure. Figure 2 presents daily median values. Samples for content of suspended solids were taken twice a week. It was demonstrated that 80–90% of suspended solids (by mass) could be consistently removed from the feed raw juice stream. Typically the residual content of suspended particles fluctuated around 400–500 mg/L. Because of high clarification temperature (about 85 °C), juice was essentially pasteurized: a significant reduction of bacterial counts was accomplished. The proposed clarification method also achieves about 10% hardness reduction. A version of this method implemented commercially in a US sugar plant has allowed some reduction in lime usage.

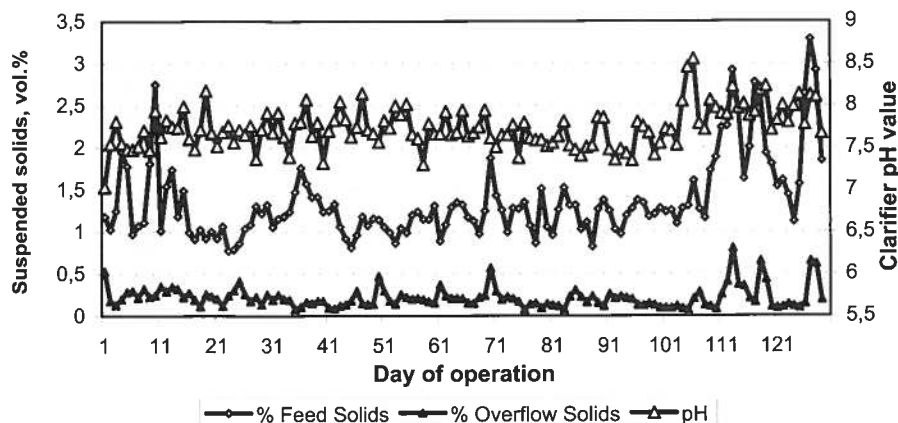


Fig. 2: Raw juice clarifier performance

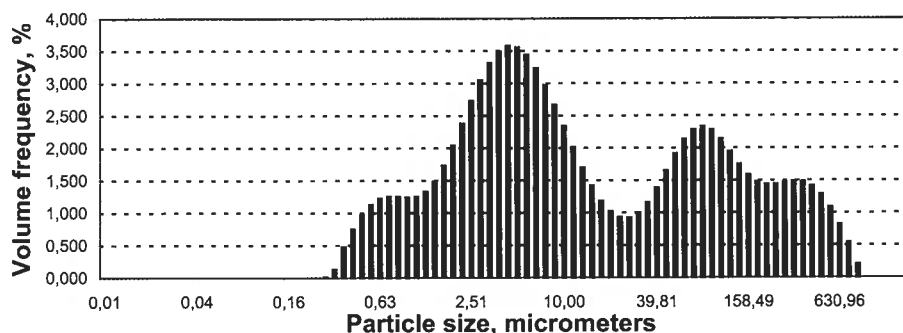


Fig. 3: Particle size distribution, clarifier overflow

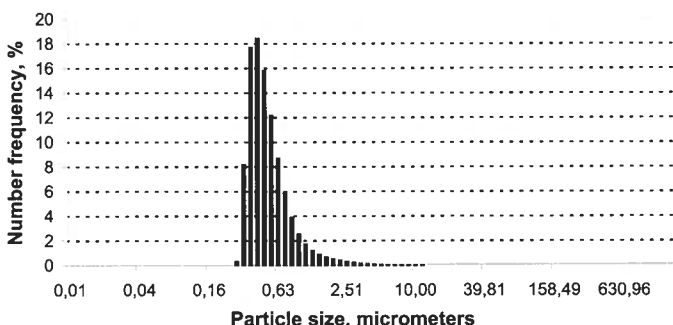


Fig. 4: Particle size distribution, clarifier overflow

A volumetric plot of particle size distribution in the clarified beet juice is presented in Figure 3. Data was obtained using a Malvern Mastersizer 2000 particle size analyzer. The plot, however, should be considered along with the data on suspended solids concentration. Presence of a few large particles skews the distribution and can greatly influence the results. It is more important to consider the size of the smallest particles present in the solution, because they will determine required cutoff for a filter. Plotting the same data set as a number distribution illustrated that most particles in the clarified juice are smaller than 1–2 μm (Fig. 4). Effective removal of particles in this size range can be accomplished using only membrane filtration.

2.3 Membrane filtration

Earlier membrane filtration work (Nielsen, 1982) targeted juice purity enhancement that would be equivalent or exceeding that in conventional juice purification. However, simple analysis of a sample raw juice composition (Table 2) indicates that this task is difficult if not impossible to accomplish using membrane filtration. Most recent membrane studies confirm that only a fraction of juice purification can be accomplished by micro- or ultrafiltration. It can be concluded that membrane filtration cannot economically replace conventional purification methods.

Studies have shown that most commercially available membranes can provide permeate of adequate quality and clarity for chromatographic purification of beet juice. However, membrane filtration of raw beet juice presents a serious challenge for membrane manufacturers for the following reasons.

1. Most sugar juices should be filtered at the highest possible temperature to reduce viscosity and prevent bacterial growth on the feed side of the membrane. It is not recommended to keep the sugar juice below 80 °C for extended periods of time. Most of the tests reported recently in the literature are performed at 85–95 °C, which presents a challenge for polymeric membrane manufacturers.
2. In beet factories the feed usually contains a certain amount of silica and sand. Although most of it is removed by beet washing, residual amounts are still present in the solution.

Table 2: Typical analysis of extract (Southern Idaho)

Component	Content		Molecular mass
	% on DS	% on nonsugars	
Sucrose	87.75	N/A	342
Invert sugars	1.03	8.59	180
Raffinose	0.42	3.5	595
Betaine	0.31	2.58	117
Citric acid	0.73	6.09	210
Malic acid	0.36	3.00	134
Lactic acid	0.12	1.00	91
Acetic acid	0.25	2.08	60
Oxalic acid	0.29	2.38	126
Other organic acids	0.20	1.67	–
Calcium, Magnesium	0.35	2.92	24–41
Sodium, Potassium	2.01	16.76	23–40
Inorganic anions (chloride, sulphate, nitrate, etc.)	2.97	24.76	less than 100
Proteins	**	–	15,000–100,000
Colorants	**	–	10,000–1,000,000
Dextrans	0.3	2.50	50,000–2,000,000
Pectins	**	–	20,000–400,000
Glutamine*	0.7	5.84	146
Other amino acids*	0.7	5.84	100–300
Unaccounted nonsugars	1.26	10.50	–
Total nonsucrose	12.00	100.00	–
Total solids	100.00	–	–

* Content of glutamine and amino acids is calculated based on molasses content of about 9% on nonsugars. ** Information was not available. *** Calcium and magnesium content is calculated based on hardness level of 12 meq/100 DS.

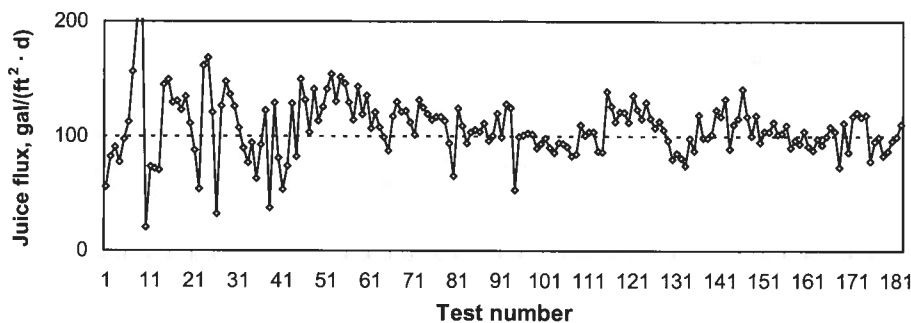


Fig. 5: Membrane performance, a total of 7,500 operating h

Being concentrated in the membrane recirculation loop at high crossflow velocities, these particles may create a danger of membrane surface erosion. In pretreated raw beet juice the concentration of acid insoluble material may reach as high as 20–30% of total suspended solids.

3. High concentration factors are required to minimize sugar losses in the retentate streams. It is not uncommon to operate a membrane system at concentration factors of 50 to 100 and use diafiltration to remove residual sugar from the remaining stream.
4. In most applications streams ranging from 300 to 700 m³/h will be filtered through membranes. Rather high permeate flux is desirable to justify the capital investment. Fluxes ranging from 50 to 400 L/(m² · h) are reported for various membrane products.
5. Composition of the solution and suspended solids content in the feed material may vary significantly during the operating season. For example, it is typical to encounter increased dextran levels in the raw juice towards the end of the beet slicing campaign. The content and quality of suspended solids also changes depending on efficiency of pretreatment operations.

A combination of the factors discussed above presents a major challenge for guaranteeing a reliable commercial operation. ARi was able to demonstrate membrane operation for 7,500 h through a period of three campaigns. A three-campaign performance graph is presented in Figure 5. Performance is presented in gallons of permeate per square foot per day; 1 gal/(ft² · d) = 1.7 L/(m² · h). Commercial size tubes and recirculation path lengths were used in ARi's pilot membrane installation to assure that results would be fully scalable. Each test lasted for 48–72 h at various concentration factors. Various cleaning strategies and operating parameters have been used throughout the test program. This explains some fluctuations in flux data, especially during the first season of operation. Flux also was found to be sensitive to juice quality; however, no good correlation with a particular juice constituent was established.

Membrane permeate quality was tested regularly throughout the test program. Permeate was always clear and free of suspended solids and bacteria. These properties make it an ideal feed stream for processes utilizing fine resin.

2.4 Softening of raw juice

Softening is a necessary step of the proposed raw juice purification process, because the presence of divalent cations (calcium and magnesium) results in deterioration of resin performance in the chromatographic process. Ion exchange softening is not a new process for sugar technologists. Thin juice softening has been used commercially for over twenty years. The process usually

benefits evaporator performance and sugar crystallization further downstream. Although the process sometimes can be justified economically based on its own merits, softening has been mostly associated with chromatographic separation technology.

Several differences exist between the composition of raw juice and molasses that affects performance of the softening process. Molasses or thin juice typically have a low level of magnesium and a high level of calcium hardness. Because of lime purification and high pH value treatment magnesium ion

is precipitated. On the contrary, about 90% of raw juice hardness consists of magnesium. Interactions of various ions in the raw juice affect the ion exchange equilibrium and result in lower resin operating capacity. Besides, raw juice typically has about 10–12 meq/100 g DS of total calcium and magnesium hardness, which is a higher value than that for molasses. Both lower operating capacity and a larger amount of hardness require a larger softening system compared to thin juice.

Depending on the type and form of resin, waste regenerant may consist of either dilute calcium/magnesium sulfate (for weak acid resin in hydrogen form) or calcium/magnesium chloride in case of strong acid resin in sodium form. Both cases have been studied at the ARi pilot plant. It has been demonstrated that a softening target of about 0.5 meq/100 g DS can be met by either of the methods. Careful evaluation of potential regenerant reuse or disposal opportunities is required for any individual factory to properly select a softening method. It is noteworthy, that raw juice hardness remains consistent throughout the processing season contrary to thin juice or molasses hardness.

2.5 Chromatographic purification studies

As a feedstock to chromatographic separator, concentrated raw juice differs from molasses in several important aspects. The ratio of sugar to nonsugars is higher than in molasses, which means that a higher solid loading is required to process an equivalent amount of nonsugars. Because ARi's chromatographic equipment is sized based on the **nonsugar loading**, the raw juice separator theoretically should be about 25% larger than the size of an equivalent molasses separator. However, use of fractal technology (Kearney, 2000) in combination with innovative control methods allows doubling the separator loading without any loss in separator performance. As a result, separator cost can be reduced by 30–40% depending on the configuration and the size of the chromatographic system. These recent improvements demonstrate that the size of existing molasses chromatographic separators is adequate to process concentrated raw juice. It is noteworthy that the same resin is used for separating either molasses or concentrated raw juice streams. Therefore, the same equipment can be designed for both services, thus increasing operating flexibility.

Recent studies confirmed that at double loading ARi's chromatographic separation technology can eliminate 85–90% of nonsugars and colorants from the feed stream. Sugar recovery across the separator exceeds 98%; extract purity consistently exceeded 97% (feed syrup purity is around 88%). Typically a higher purity of extract can be reached if feed purity is higher.

2.6 Sugar end comments

It was demonstrated in earlier studies that white sugar satisfying or exceeding current specifications could be crystallized from chromatographic extract. Because of low extract color (typically below 1000 ICUMSA units) it is expected that at least two strikes of white sugar can be obtained in consecutive crystallization stages. Due to the high purity of chromatographic extract, the factory sugar end may need to be modified to ensure complete molasses exhaustion. Solubility studies indicate that molasses purity as low as 43% can be expected. The material balance calculations show that more crystallization stages are required to reach target molasses purity compared to a conventional factory. However, no additional equipment will be needed due to relatively small amounts of high and low raw sugars. In a separate study Vaccari et al. (2001) also demonstrated that two crops of white sugar could be obtained from chromatographic extract using cooling crystallization.

3 Economic aspects

A complete economic evaluation taking into account existing equipment must be performed for each individual plant in order to estimate the benefits of the raw juice purification process. An economic analysis was approached by breaking down the project into two tasks. The first task includes membrane filtration and related pretreatment. As was discussed earlier, membrane filtration alone does not provide the benefits necessary to justify the purchase of a large-scale system. It was attempted to analyze, whether membrane technology could replace conventional liming and carbonation assuming that it is complemented by chromatographic purification and softening. Molasses chromatographic desugarization had already been proven to be feasible for the US sugar industry. Therefore, it is necessary to show that the use of membrane filtration in the beet end can provide savings sufficient to justify the capital expense.

The following assumptions proved to be useful for the analysis.

1. The chromatographic separator can be easily converted from molasses feed to softened and filtered raw juice.
2. The chromatographic separator can be economically justified based on extraction benefits.
3. Raw juice softening and additional evaporation requirements are included in the separator justification.
4. Changes in the sugar end are not included in the analysis, since they are specific for each plant.
5. The maintenance and operating cost estimates for the membrane system are taken from a quote provided by an equipment manufacturer. The quote was based experimental data provided by ARI.

Table 3: Materials eliminated or added in the beet end

Eliminated

- Lime rock and spalls
- Kiln fuel (coke or natural gas)
- Screened coke
- Soda ash
- Filter cloth for thin juice filters

Reduced

- Filter-aid
- Defoamers and flocculating agents

Added

- Chemicals for membrane cleaning and pH value adjustment
- Lime for flume pH value adjustment

Table 4: Expected changes in operating expenses

Expenses	Reduction in %
Cost of materials and chemicals	67
Maintenance and repair*	55
Power usage	20
Environmental cost related to lime kiln and lime ponds operation	100
Labor	22
Membrane replacement	Added cost

* Maintenance cost for a membrane system was estimated at 2.5% of total capital cost according to recommendations of a system manufacturer.

It was thoroughly studied which areas of the beet end would be affected by the installation of a membrane filtration system. The largest contributors are listed in the Table 3. The estimated annual changes in operating cost are reflected in Table 4. Membrane replacement will be the major contributor to the operating cost in the new lime-free purification process. The annual replacement cost depends on membrane selection. For the analysis the annualized cost of ceramic membranes under the assumption of five years membrane lifetime was used. The replacement cost would be similar for polymeric membranes with a lifetime of one campaign. The overall operating cost of a conventional beet end in a 7,000 t per day slice rate factory for a 160-day campaign was estimated at \$4,180,000. This number includes cost of materials, labor, power and maintenance.

Similar calculations for a beet end including a membrane system showed an expected operating cost of \$2,366,000. This number includes the annualized membrane replacement cost. The resulting savings would be \$1,814,000 per year or \$1.62 per ton of beets. These calculations can be included in the overall process feasibility study for raw juice purification technology. The cost of membrane filtration, softening and chromatography and other information required for such study is now available from ARI.

4 Roads to implementation

Since the proposed process affects many areas of an existing plant, the process will be most likely implemented stepwise. Several strategies of implementation may be considered.

1. Molasses desugarization may be a reasonable first step to implement the new process. Processing of a partial stream is also possible depending on the size of the system. Equipment can be later converted to process concentrated raw juice.
2. Membrane filtration and softening can be useful if a part of the concentrated raw juice is stored till after the processing season. The storability of concentrated raw juice has been confirmed in separate tests on a pilot scale. This option may be useful for factories with sufficient slicing capacity but limited purification capability. Treating a partial stream of raw juice using clarification and membrane filtration may help increase processing capacity by 10–20%.
3. The operational flexibility of chromatographic separators allows varying the purity of the extract stream. Lowering extract purity is usually accompanied by increased sugar recovery, and may be used to minimize changes in the sugar end.
4. For companies operating several plants, transporting concentrated raw juice (rather than beets) may be helpful to reduce transport costs. Other alternatives may also be considered. Additionally, a company may take advantage of the features of chromatographic raw juice purification highlighted in the following section.

5 Benefits of the raw juice purification process

1. There is a complete elimination of liming and carbonation steps from beet sugar production process. Some of the existing equipment (clarifier, tanks, pumps, controls) may be used in the new process. Problems related to lime cake disposal, lime kiln and carbonation operation and maintenance are completely eliminated.
2. The total amount of solid waste is reduced by a factor of seven to eight (depending on the location and factory current lime usage). The solid waste product consists of beet particles and residual soil. Because of residence time in the clarifier at 85 °C bacterial counts are very low. The studies have showed that the clarifier product has certain nutritional value as a cattle feed, but cannot compete with other conventional feedstock. The solid product can also be composted and returned to the field.
3. There is a very high level of nonsugar elimination across the chromatographic separation (85–90% compared with about 30% in the conventional process). Some of the nonsugars that are difficult or impossible to remove using conventional purification are eliminated or significantly reduced.
4. There is the potential for storage of the sterile micro filtered syrup. The quality of stored raw juice becomes consistent.
5. The chromatographic separator can be used in a variable manner and process either concentrated raw juice or molasses depending on factory scheduling.
6. The process is compatible with various sugar end configurations, including cooling crystallization of chromatographic extract.

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7. Odors generally associated with white sugar are reduced by the new process because the compounds ordinarily formed by liming and carbonation are eliminated.

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Épuration chromatographique du jus brut de betteraves. Une technologie sans faire usage de chaux (Résumé)

Un procédé d'épuration du jus brut de betteraves ou de cannes par traitement chromatographique a été proposé et breveté par l'Amalgamated Research Inc. (USA) il y a quelques années. Dans son application aux betteraves, ce procédé comporte une élimination des particules solides en suspension dans le jus brut avec adoucissement ultérieur, évaporation et épuration chromatographique du sirop obtenu. Comparativement à l'épuration classique, le procédé chromatographique assure une élimination significativement plus élevée du non-sucre et de la coloration sans faire aucun usage de chaux. L'utilisation d'appareils de distribution fractale de fluides a augmenté notablement l'efficacité du procédé proposé en permettant une diminution sensible des consommations dans les procédés de séparation par résines. La possibilité d'obtenir par cristallisation un sucre blanc de haute qualité a été démontrée par plusieurs essais indépendants. Ce procédé procure en général des rendements d'extraction supérieurs à 92 %.

Purificación cromatográfica sin cal del jugo crudo de remolachas (Resumen)

Algunos años atrás, la compañía Amalgamated Research Inc., E.E.U.U., recomendó y patentó un método para la purificación cromatográfica de jugos crudos de remolachas y de caña de azúcar. Para la industria azucarera de remolachas el método incluye: separación de sólidos suspendidos en el jugo crudo y luego endulzamiento, evaporación y purificación cromatográfica de las mieles. Comparado con la purificación tradicional del jugo, este método cromatográfico permite eliminar mayores cantidades de no-azúcares y de colorantes sin el empleo de cal. Distribuciones fractales de líquidos mejoran significativamente la eficacia del método, ya que es posible reducir considerablemente las cantidades de las sustancias en las columnas de intercambio iónico. En varios ensayos independientes se demostró la cristallización de azúcar blanco de alta calidad. El rendimiento de azúcar con este método es de sobre 92 %.

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