SMB Chromatography as an Alternative to the Conventional Refining of Raw Cane Sugar
#1268

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Abstract. Simulated moving bed (SMB) chromatography is a well-established and widespread unit operation in the beet sugar industry and has been practiced commercially for more than thirty years. SMB chromatography has proven to be very efficient at removing inorganic ash components and colorants from sugar liquors, particularly those colorants with relatively high molecular weights. Recent optimization of the separation using raw sugar with a color ranging from 2,000 IU up to 4,000 IU has focused on minimizing the capital and operating costs of a chromatography system to replace the various purification and decolorization steps in conventional raw cane sugar refining. Decolorization levels exceeding 89% have been reliably achieved in the extract fraction from chromatography, along with ash removals ranging from 92% to 97%. As only water is added to purify the sugar, the application of this technology, in combination with modern industrial heat pumps and mechanical vapor recompression, allows for a powerful CO_2-neutral process, with all the associated benefits regarding permits and marketability. The potential of this technology includes integration into new refinery projects (either standalone or back-end) by replacing affination, carbonatation and ion exchange decolorization (with raw sugar colors of up to 5,000 IU), to expansion of existing refinery sites for slip stream processing of melted raws or refinery run-off clarification, and to the introduction of new process steps, such as direct liquid sugar production from cane raw sugar.

Introduction. The main purpose of cane refineries is to produce sugar that has high purity in sucrose and is free from impurities such as ash and color molecules. Removal of color is one of the most important functions of a refinery (Rein, 2017). The color of the raw cane sugar and the color specification of the product white sugar generally influence what processes are incorporated into a cane sugar refinery. The choice of the optimal refinery decolorization scheme depends on various factors, but ultimately the goal of the decolorization process is to obtain a syrup of a color that, when processed in the pans, would reliably produce final sugar color that complies with the specifications for white sugar, utilizing the desired number of crystallization steps (Vawda, 2014).

In 2010 *Amalgamated Research LLC* (*ARi*) developed and patented a method for removal of impurities from a high-purity sucrose material, including raw cane sugar (Costesso *et al.*, 2011). The method used chromatography to purify the high-purity sucrose material from relatively small quantities of non-sucrose impurities such as ash and color components (Costesso *et al.*, 2011). The research work carried out by *ARi* used an eight-cell chromatography system to process a feed syrup obtained from raw cane sugar, achieving about 70% or more color elimination, and resulting in a product with high sucrose purity. The quality of the sugar stream was improved such that substantial amounts of ash components were removed as well.

The sugarcane industry uses non-chromatographic methods such as affination, carbonatation, and phosphatation to purify raw cane sugar prior refining through crystallization. Chromatography is a technology widely spread in the sugar beet and corn industries, where is used to purify target components from valuable feedstocks but has not been widely applied in the sugar cane industry. Costesso *et al*. suggest that the patented method may be applicable to purify a wide number of high-sucrose containing materials such as raw cane sugar, turbinado, demerara, muscovado, or brown sugars. Also, Kochergin (2000) suggests that the cane sugar industry could benefit from applying chromatography to other process streams. Previous research at *ARi* demonstrated that producing white sugar directly in a cane mill from clarified juice is technically feasible, increasing sugar mill recovery by 6.8% (Kochergin *et al*., 1999).

Recent research at *ARi* has found that SMB chromatography can be used to purify raw sugar of a relatively high color (for example from 2,000 IU – 4,000 IU). The result is a sugar-rich stream with a purity above 99%, with elimination of more than 97% of the inorganic ash components (measured as conductivity ash) and removal of 89% of the initial color (measured as ICUMSA units). This method for purifying raw cane syrup offers several advantages, such as the elimination of chemical consumption, as only water is needed for dilution and elution purposes. The water used in chromatography can readily be recovered from the extract and raffinate streams by energy-efficient evaporation. This recent optimization has the potential to replace the various purification and decolorization steps in conventional raw cane sugar refining.

The following sections include the results of the pilot work carried out, as well as a discussion of different process configurations in which chromatography could be used as an alternative to traditional refining technologies.

Pilot-Scale SMB Chromatography.

Pilot-Scale Chromatography Testing. Simulated moving bed (SMB) chromatography is a well-known unit operation that has been applied in the beet sugar industry for more than thirty years. In beet sugar applications where molasses desugarization systems are used, sucrose is separated from non-sugars. A general process flow of this two-component separation is shown in Figure 1. The feed is separated into two product streams: the sucrose-rich extract, which is sent to the sugar end for crystallization, and the sucrose-lean raffinate, which includes color and salts. The raffinate can be used as cattle feed (Kochergin *et al*., 2019).

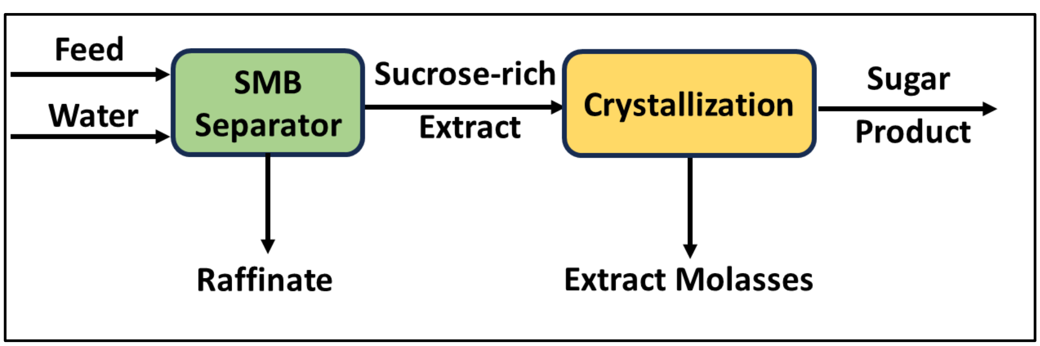

Figure 1. Process flow of a molasses desugarization system separating two components

Chromatography is a method that allows the separation of components in a mixture, based on the different interactions between the stationary phase (resin or adsorbent) and the mobile phase (feed material). The capacity of a chromatographic separator can be represented through the loading, which quantifies the amount of feed material that can be processed through a system of a given volume of resin in a given period of time (Kochergin *et al.*, 2019). The maximum achievable system loading determines the column diameters, the amount of resin required, throughput, and therefore the capital cost of a system to carry out a specific separation or purification process.

The eluent, in this case water, provides the mobile phase that allows the movement of the various soluble components through the column. The inputs to a system for processing raw cane sugar would therefore be the feed material (raw sugar syrup) and water. The expected products would be a sucrose-rich extract stream, low in color and inorganic ash components, and a raffinate stream rich in ash and color molecules. Experimental pilot work was carried out at the *ARi* pilot plant facility in Twin Falls, Idaho. The feedstock comprised of two samples of raw cane sugar of characteristics provided in Table 1. Each batch of feed liquor was made by dissolving raw cane sugar in deionized water to a final dissolved solids content of 65 – 67% and filtering it to remove suspended solids.

Table 1. Initial analysis of raw cane sugar samples

Sample [-]	**Polarization** [°Z]	**Color** [IU]	**Turbidity** [IU]	**Conductivity Ash** [%]
A	98.5	1,947	1,289	0.119
B	98.0	3,649	1,297	0.362

The separation efficiency of components in chromatography in the sugar industry is dependent on the chromatography resin remaining in a monovalent cationic form. Therefore, calcium and magnesium need to be removed from the syrup before the chromatography operation, as these ions would convert the ionic form of the strong acid cation exchange resin used (in this case, *Mitsubishi Diaion UBK522M*). Due to the initial high divalent-ion content in the feed liquor obtained from raw sugar sample B, a column filled with a strong cation resin was used for softening of the feed liquor before chromatography, lowering the divalent cation concentration of the feed to below 1.0 mEq/100 g dissolved solids.

SMB chromatography of the pretreated raw sugar liquor was carried out at 75 ^{o}C in a pilot system consisting of six columns in a series arrangement, each of nominal diameter of 2 inches (50.8 mm) and with a bed depth of 5 feet (1.5 m). The total volume of resin within this pilot system was 18.5 liters.

Pilot testing was performed following *ARi*'s formalized optimization methodology for SMB chromatography systems, by locating the operating parameters that allow for an optimum separation, while minimizing the capital and operating costs of the process. The data obtained from this testing is typically used to generate contour plots that show the response of the system performance metrics (such as sucrose purity, color and sucrose recovery) to variations in the system operating parameters.

Pilot-Scale SMB Chromatography Results. Typical results obtained when processing the two raw sugar feedstocks (A and B) are shown in Tables 2 and 3. This data suggests that SMB chromatography is a technically viable alternative to more-traditional methods of raw cane sugar refining. Relatively high levels of color elimination were achieved, when compared to other available technologies. The inorganic ash components originally present in the raw sugar were successfully removed, at levels of up to 97%. Turbidity content showed a similar trend, where the levels in the product fraction were reduced between 80% to 93%, demonstrating that chromatography is very efficient at generating an extract product with characteristics suitable for the production of white sugar.

Table 2. Optimization results for Case A feed syrup

SMB Streams [-]	**Dissolved Solids** [%]	**Color** [IU]	**Sucrose Apparent Purity** [%]	**Sucrose Extract Recovery** [%]	**Conductivity Ash** [%]	**Turbidity** [IU]
Feed	67.0	2,018	98.71	99.13	0.120	1,289
Extract	43.1	341	99.05		0.008	94
Raffinate	0.9	74,234	42.16		0.085	56,085

Table 3. Optimization results for Case B feed syrup

SMB Streams [-]	**Dissolved Solids** [%]	**Color** [IU]	**Sucrose Purity by HPLC** [%]	**Sucrose Extract Recovery** [%]	**Conductivity Ash** [%]	**Turbidity** [IU]
Feed	64.9	3,649	98.00	99.36	0.362	1,297
Extract	39.0	415	99.41		0.011	218
Raffinate	1.0	117,002	27.13		0.267	31,023

Examples of contour plots for some chromatography performance metrics are shown in Figure 2. In these examples, raffinate sucrose purity and color elimination were chosen. These parameters are shown as functions of the system loading and water consumption, which are critical factors influencing the capital and operating costs of a chromatography system. The contour plots are interpreted by locating the desired performance target and then reading off the maximum loading and minimum water consumption achievable while still meeting the performance metrics. The contour plots generated by *ARi* provide a full characterization of the chromatography system performance as a function of its operating parameters. They can also be used for sensitivity analysis.

The difference between the operating parameters recently developed during the present pilot-scale work, using a 6-cell separator, and past operating settings (100% baseline) developed utilizing a 8-cell separator, is shown in Figure 2 in terms of percentages. For instance, to generate an extract product fraction with an 83% color elimination, coupled with a raffinate stream where the sucrose purity is no greater than 25%, the chromatographic system would have a capacity 29% greater than previous sizing parameters (intersection of dotted red lines in Figure 2, left-side), while the water consumption would be 2% lower compared to previous available data (intersection of dotted red lines in Figure 2, right-side).

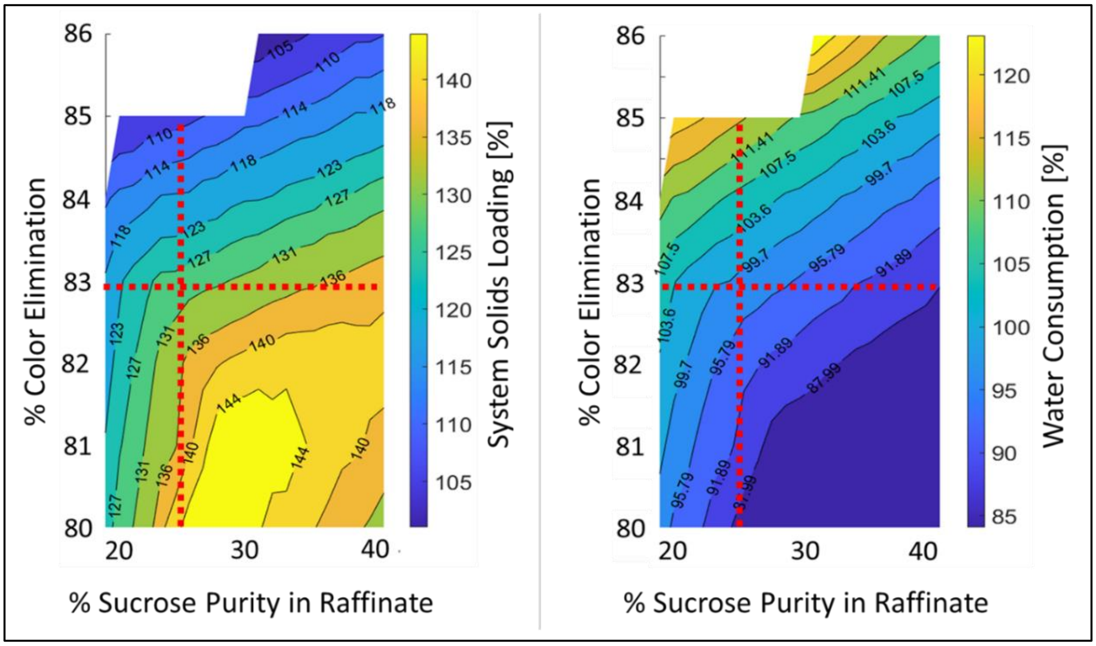

Figure 2. Contour plots obtained with a feed liquor of a color of 4,000 IU.

Chromatography as an Alternative to Conventional Refining of Raw Sugar. Chromatography proved to be a robust technology that can treat various feedstocks derived from cane sugar enabling significant process flexibility. Chromatography can be integrated into expansion projects by means of slip stream processing of melted raws or refinery run-off clarification. This technology can also be integrated into the refining process via introduction of new process steps, such as direct liquid sugar production from cane raw sugar. For the purpose of the current paper, a focus has been placed on the use of SMB chromatography technology in a back-end refinery associated with a raw cane mill. While SMB chromatography can also be applied to a standalone refinery configuration, it holds potential additional benefits when integrated into back-end refining and these are highlighted.

Back-End Refining. A portion of a generic process flow diagram for the refining of raw sugar in a back-end refinery using SMB chromatography is shown in Figure 3. In this process configuration, the sugar obtained from the rawhouse is melted to a dissolved solids concentration of 67% and then filtered to remove any suspended solids. Depending on the composition of the ash components in the filtered raw liquor, it may need to be softened prior to chromatography. The pretreated liquor would then be fed into the separator and the sucrose is purified through SMB chromatography, with the addition of water as eluent. Two streams are obtained in this process: a stream rich in sucrose, low in ash and color, high in dissolved solids, known as extract, and an impurity-rich stream, high in color and ash, low in dissolved solids, known as raffinate. After chromatography, the product streams are concentrated through energy-efficient multiple effect evaporation, with the recovery of water as condensate. The condensate from the evaporation operation is re-used as elution water in the chromatography process. The concentrated extract stream proceeds to refined sugar crystallization.

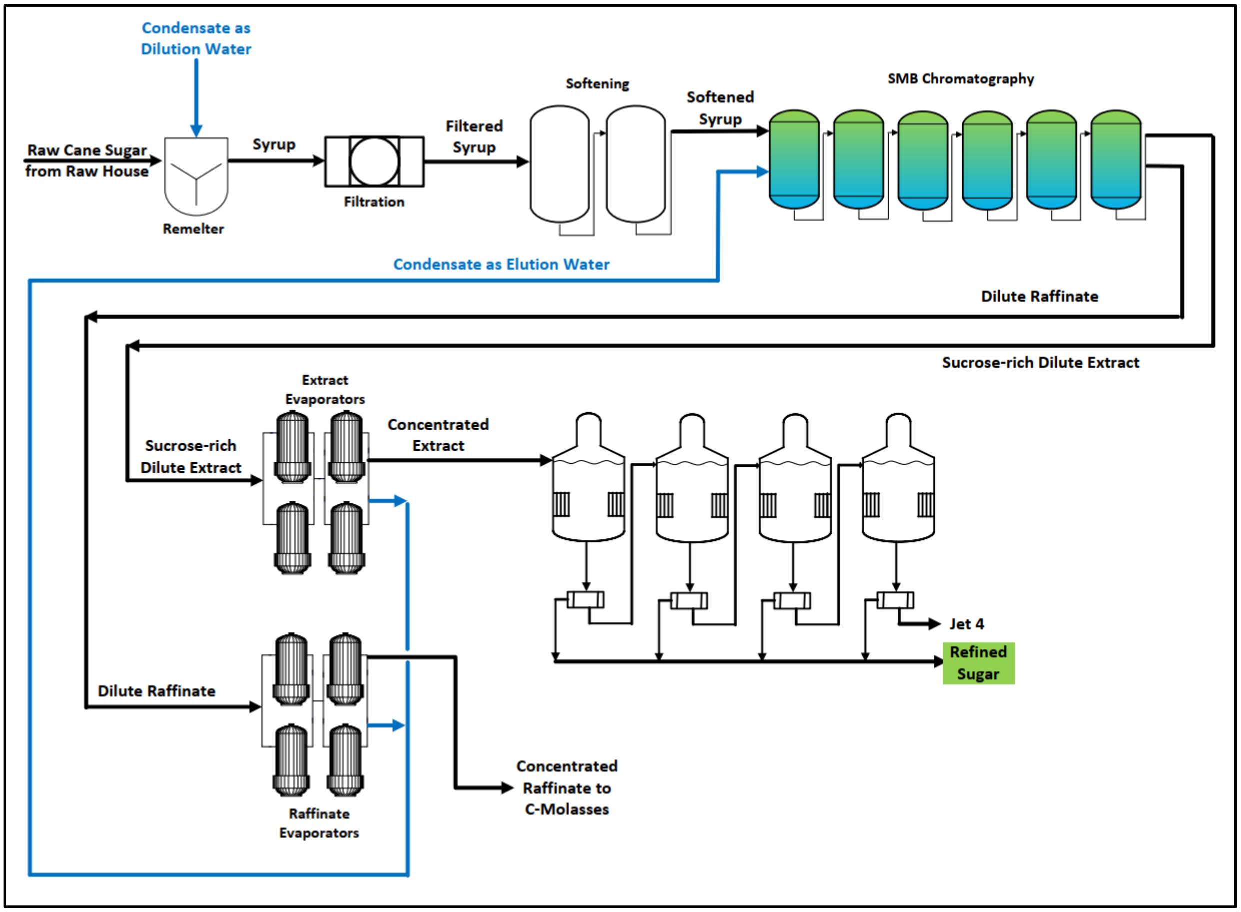

Figure 3. Potential flow sheet for the back-end refining of raw sugar using SMB chromatography.

In a back-end refinery, the processing scheme in the rawhouse can be tailored to suit the technology employed in the refinery. An advantage of using SMB chromatography in the refining process is that it can tolerate a raw sugar feed of relatively low quality, while still producing an extract liquor that is suitable for white sugar crystallization. The associated rawhouse can take advantage of this by producing a relatively low-quality raw sugar, thereby minimizing capital costs and energy consumption.

Typically, a rawhouse with a back-end refinery would produce sugar at VHP quality (or better), in order to minimize the difficulty of refining the raw sugar to produce white sugar product at a suitable quality. This increases the equipment needed to produce this raw sugar product, as well as the energy demand and operating costs involved. By contrast, a back-end refinery using SMB chromatography could tolerate a raw sugar color as high as 4,000 IU or more. This allows for a lower-cost raw pan house configuration, along with savings in energy and an increase in sucrose recovery due to reduced recycle and lower molasses purity within the factory.

In Figure 4, a modification to the VHP boiling scheme in the rawhouse is proposed. Both the A- and B-boilings are seeded with magma derived from the C-sugar, and the product sugar from both of these boilings is combined together for processing in the back-end refinery. As the need for producing a high-quality raw sugar product no longer exists, it would not be necessary to use any high-purity syrup in the B-boiling, as would usually be required when both the A- and B-sugars are mixed to produce a final product. The usual recycle of B-sugar through the panhouse is avoided, thereby reducing the equipment requirements for this process, as well as its associated energy consumption. An additional benefit is that some of the non-sugar impurities in the process leave the factory in the chromatography raffinate stream at a very low purity of about 20%. This effectively reduces the ultimate loss of sucrose to final molasses and thus increases the overall sucrose recovery in the process.

In this model, the mixed raw sugar to the refinery would be expected to have a color of approximately 3,500 IU, which can readily be processed into a chromatographic extract fraction of below 400 IU in the back-end refinery. After evaporation, the extract liquor could be used to generate four white sugar product boilings, with a mixed refined sugar color of 40 – 45 IU. This refined sugar product would be expected to comply with the typical specifications for a refined sugar product.

***Product Quality and Process Opportunities*.** As defined by Vawda *et al*, (2014), process flexibility refers to the capacity of a process to handle expected and unexpected disruptions in the operational demand, in this case, the chromatography process was found to have substantial operational flexibility, by being able to process various raw sugar feedstocks of significantly different qualities while still producing a high-quality product fraction. In this study, feedstocks of color ranging from 2,000 IU to 4,000 IU were treated with SMB chromatography achieving color removal up to 89%, showing that chromatography has the potential to outperform other technologies traditionally used in the decolorization of raw cane sugar. This flexibility may be of value to the refiner, when faced with the challenge of processing varying raw sugar qualities during the crop year and when the increasing color levels of the raw sugar in storage demand more adaptability in the refining process. An additional benefit of using chromatography in the refining of raw sugar is found in the potential reduction of fouling and/or scaling of the evaporators and pans due to the softening pretreatment of the liquor before chromatography.

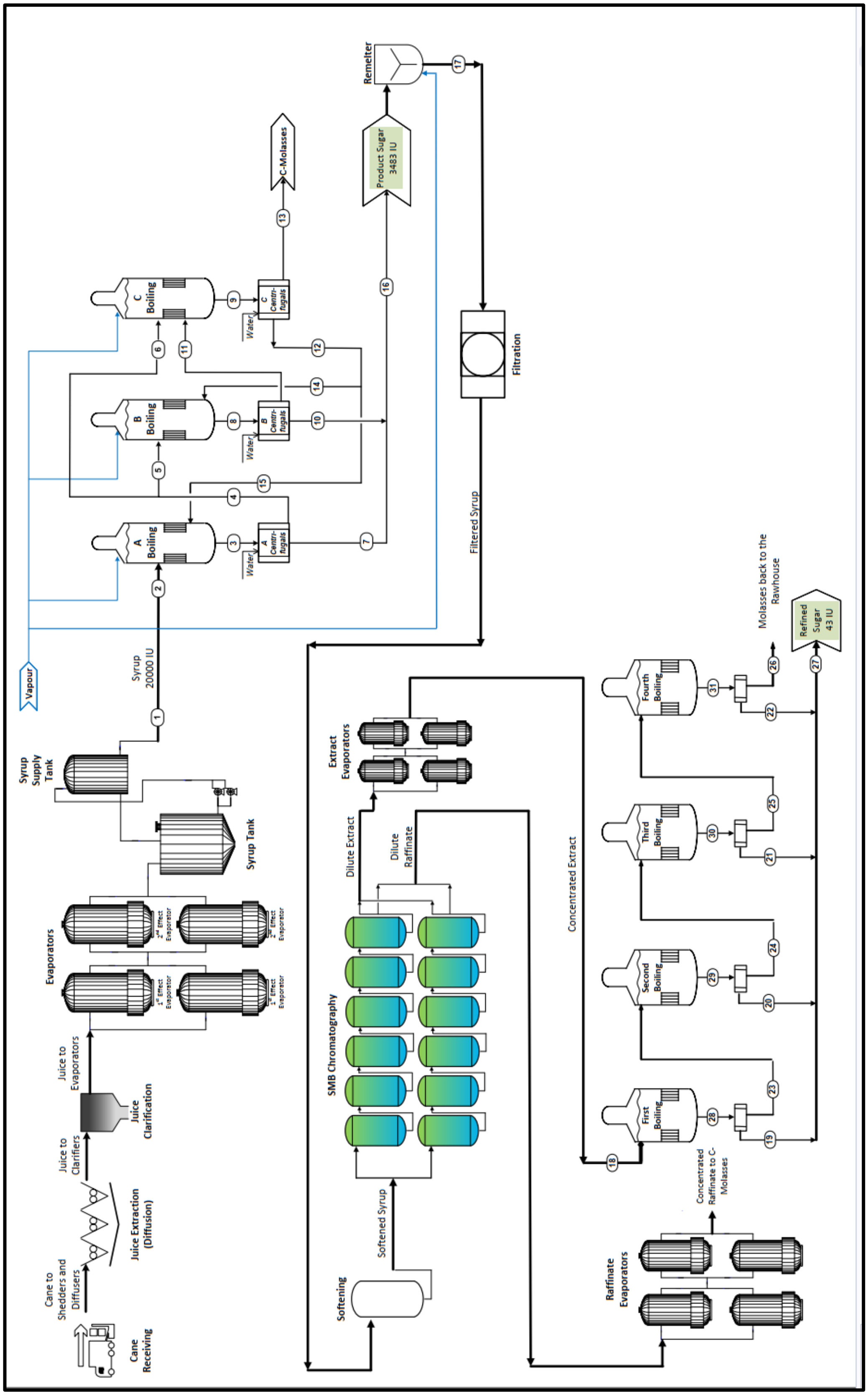

Figure 4. Flow sheet of the cane sugar refining -back-end refinery

Vawda *et al*, (2014) indicate that common refinery decolorization methods relying on activated carbon typically result in color removal of 80% to 85%, while ion exchange resins coupled with phosphatation can achieve color reductions of 60% to 70%. When coupled with carbonatation, ion exchange typically achieves 70% to 75% overall decolorization. Coterillo (2023) reports up to 80% color removal through ion exchange resin columns (IER) of cane liquor in a sugar refinery plant, while Charbonneau *et al.* (2022) also report 80% as color elimination in a cane sugar refining application. The results from the present pilot testing showed that the color elimination achieved through chromatography ranges from 84% to 89%, outperforming most conventional technologies.

Tuson (1961) states that the removal of color and ash are inseparable topics that impact the refining processes, and final product quality. Cations like sodium and potassium significantly affect the yield, whereas sulfates and carbonates can deposit on the surface of pans, as calcium salts, requiring periodical boil outs to recover heat transfer efficiency. Typically, where it is used, affination is the main operation for ash removal in the cane sugar industry (Rein, 2017), while carbonatation and bone char can remove the small remaining amounts of ash from affined sugar. Rein (2017) reports roughly 10% to 20% removal of ash from cane syrup using carbonatation. The present pilot work conducted showed that chromatography can serve as a potential replacement for the processes traditionally used for de-ashing in the refining of raw sugar with levels of removal up to 97%.

Turbidity is another important quality parameter that affects the final product specifications. Lima *et al* (2023) report turbidity removal levels of 42% on average when using powder activated carbon (PAC) on sugarcane refinery melt liquor in a pilot plant. Refiners use clarification methods such as carbonatation and phosphatation to remove color and turbidity, Lengen *et al* (2022) report turbidity reduction by 73% when using adsorbent media in conjunction with phosphatation to treat processed raw cane sugar, and >60% when using phosphatation alone (Lengen *et al* (2019). While the industry is looking for alternative options for decolorization and impurity removal such as the introduction of more effective adsorbents (HPA) coupled with traditional decolorization operations, the results from this research indicate that chromatography allows for turbidity reduction ranging from 84% to 93%, placing this technology as a viable option for the refining of raw cane syrups.

Crystallization studies carried out by Kochergin *et al.* (2000) showed that extract samples obtained through a chromatographic process could be boiled to produce a white sugar of high quality and low color. Since chromatography is very effective at removing colorants of higher molecular weight, the color transfer from mother liquor (derived from SMB extract) to sugar during crystallization is typically reduced. As a result, the extract colors of 300 – 400 produced in the current study would be expected to yield very low sugar colors upon crystallization. Further research needs to be done to study the possibility of including a 5th white sugar product boiling that could lead to an increase in the overall sugar recovery, based on the expected reduction in color transfer from product treated with chromatography. Further crystallization studies using SMB extracts obtained from raw sugar processing through chromatography could potentially be investigated in future research, to assess the final quality of the white sugar produced using this technology.

In addition to the traditional production of crystalline white sugar as a product, the use of SMB chromatography in refining also brings more process opportunities as it facilitates the production of a high-quality liquid sugar product. As part of the pilot testing at *ARi*, the sucrose-rich extract stream from chromatography was further decolorized using granular activated carbon (GAC). Extract with an initial color of 300 – 340 IU was readily decolorized to generate a product liquor with a color of 12 – 15 IU, with sucrose purity of 99.2% and conductivity ash levels of 0.009%. These pilot-scale results indicate the technical feasibility of obtaining a product stream satisfying the typical liquid sugar specifications. Figure 5 shows samples of the various process streams (feed, extract, liquid sugar) obtained through chromatography followed by polishing with GAC.

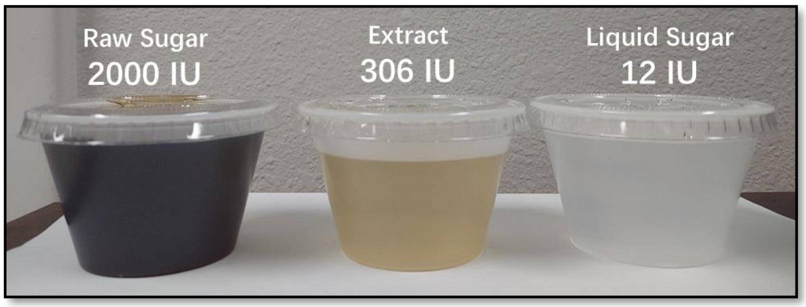

Figure 5. Samples of various process streams through chromatography and polishing steps

Sustainability Opportunities**.** It is a common trend that more cane sugar end-users, or buyers, are looking into acquiring goods whose production is sustainable. Cane sugar producers are increasing their efforts to reduce greenhouse gas (GHG) emissions generated during the refining of sugar to align their operations with production standards such as *BonSucro*. For new or existing plants, the environmental regulations are becoming stricter, and carbon footprint monitoring is becoming the norm to track factory inputs in climate change issues -carbon footprint measured as total GHG emissions directly or indirectly generated by the industrial processes of an organization, Cabrera (2011)-.

As stated by Vawda *et al.*, (2014) technologies such as bone char and GAC have significant environmental impact, due the utilization of thermal regeneration performed in kilns that generate emissions. In the case of chromatography, the adsorbent does not require regeneration of any type. This represents an advantage over other processes. The adsorbent media recovers its ability to remove color rapidly as the interactions between the resin particles, sucrose, colorants and ash molecules are of a weak nature unlike GAC or ion exchange. Chromatography is an alternative in the refining of raw sugar, improving the environmental impact of sugarcane processing compared to operations such as liming or carbonatation. The latter processes present serious disadvantages in terms of waste generation and disposal. The introduction of SMB chromatography eliminates the need for liming, carbonatation and affination operations in the refining of raw sugar, therefore the issues associated with waste disposal, maintenance and operation of these units can be minimized by chromatography.

Carbon footprint also relates to emissions associated to waste generation and disposal. Chromatography only uses water as main input for the operation; therefore, no direct waste generation is associated with the separators where non-harmful effluent streams are produced, adding to the refineries efforts to responsibly reduce and manage waste streams. Furthermore, the elution and dilution water necessary for the chromatographic process can be effectively recycle within factory. Energy management can be applied to the evaporation of chromatography streams by implementing energy efficient configuration schemes such as low-pressure steam generation using waste heat, and mechanical vapor recompression.

Economic Aspects. A material balance based on a mass flow of incoming syrup to the rawhouse of about 140 t/h (at 67% DS) was performed around the SMB chromatography step, evaporators, and pan floor using inputs from this pilot study and parameters from traditional boiling schemes in the sugarcane industry. Although, a full economic evaluation must be conducted on the proposed process, rough calculations were performed to estimate initial economics. Preliminary estimates suggest some cost saving opportunities: the modified boiling scheme offers potential savings in terms of equipment requirements, as well as operating costs represented by the steam consumption for each scenario. As shown in Table 4, a reduction of 18% in the massecuite volume processed in all boilings, that directly relates to pan sizing, is expected when producing a low-quality raw sugar that will then be processed with SMB chromatography. Additionally, the steam requirements in the modified boiling scheme are reduced by 25% compared to the traditional VHP scheme, due to less material recycle in the process.

Table 4. Results from modeling a raw sugar factory by introducing a back-end refinery step that uses SMB chromatography for the refining of sugar

Stream		**Conventional VHP scheme**	**Modified boiling scheme to produce low quality raw sugar**
Volumetric flow of massecuite	[m^3/h]	145	123
Vapor consumption	[t/h]	94.2	75.5
Raw sugar, sucrose mass flow	[t/h]	72.8	73.5

An important economic issue associated with the use of SMB chromatography is the dilution of the product streams. The cost of concentrating the extract and raffinate product streams to a dissolved solids content that is appropriate for further processing is the major operating cost of the chromatography process. As an example, a material balance was conducted for a back-end refinery processing 3,347 tons per day of raw sugar syrup at 67% dissolved solids. The total steam requirement for the evaporation of the extract and raffinate streams from chromatography was determined to be ~85,000 lbs/h of steam requirements (LP steam, 20 psig). Although around half of that steam requirement would be saved in the rawhouse due to the reduced steam consumption associated with the modified boiling scheme that has been proposed, it would still be necessary to supply the factory with an additional 20 t/h of steam – either through the purchase of additional fuel or by increased energy efficiency measures in the rawhouse to allow for the supply of this steam requirement using the existing bagasse supply.

As mentioned earlier, the introduction of the SMB chromatography process allows some of the non-sugar impurities in the process to be eliminated in the raffinate waste stream, at a very low sucrose purity of about 20%. This effectively lowers the overall loss of sucrose to final molasses (as the blend of raffinate and final molasses leaving the plant is at a lower purity than normal) and increases the overall recovery of the factory. Modelling of the rawhouse (attached to a back-end refinery that uses chromatography) has shown an overall recovery of 91.5% to be achievable, compared to a value of 90.8% when using a conventional VHP boiling scheme (attached to a traditional back-end refinery) - both of these recovery figures, determined by means of modelling, exclude any undetermined losses in the factory and are thus slightly higher than the values usually achieved in practice-. This increase in recovery represents an additional 2,500 tons per year of refined sugar production for a factory and back-end refinery operating for 4,000 hours per year. At a conservative estimate of 30 US cents per pound of refined sugar (based on the *ICE White Sugar #5* – 2023 Sugar price), this would yield an additional $ 1,6 million per year to the refinery (which would exceed the expected fuel cost for the additional steam required by the use of SMB chromatography). Additional recovery benefits would be achievable if a fifth refined sugar boiling could be bagged, due to the lower color transfer expected when crystallizing from an extract stream generated using chromatography.

Conclusion. SMB chromatography is a well-established technology that presents an alternative to the traditional refining schemes in the cane industry, by generating a product stream of high-purity, low ash content and low color that can be crystallized in a conventional pan floor. Preliminary mass and energy balances indicate that chromatography offers several advantages from process, product quality and sustainability standpoint. Using chromatography for the refining of raw cane syrups enables color elimination above 89%, de-ashing removal up to 97%, and removal of turbidity up to 93%. This study serves as the basis for further research that incorporates a holistic approach to include steps like crystallization of the extract for color transfer studies, as well as further optimization of syrups with lower solids concentration.

Acknowledgements. The authors would like to acknowledge Christopher Dayton and Keith Vinecke, for their highly valuable contributions during the execution of the experimental pilot work conducted at the *Amalgamated Research* facility.

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