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in Cane Mills using
Chromatographic Purification of Clarified Juice**

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Abstract

A process for production of white sugar directly from clarified cane juice developed and patented by Amalgamated Research Inc. (ARi) is presented. Chromatographic separation proved to be efficient in separating cane non-sugars, including ash and invert, from sucrose. A search for an economically feasible method of suspended solids removal from clarified juice is in progress. The challenges facing the membrane manufacturers are discussed. The samples of clarified cane juice have been filtered, softened and concentrated prior to chromatography. Application of ARi's coupled loop chromatography method allows low color extract with very low ash and invert levels to be obtained. Subsequent crystallization in a continuous 60-liter vacuum pan has yielded sugar crystals meeting all requirements of refined white sugar.

Keywords: clarified juice, chromatography, membranes, white sugar

For years raw sugar production was the only feasible option for a cane mill to operate independently from a refinery. Unlike a beet sugar plant, where raw juice undergoes at least two filtration steps before crystallization, cane raw juice is only subjected to clarification or flotation. Although the cane juice purification process efficiently removes suspended solids, it does not significantly affect elimination of the large molecular weight dissolved components. It is well known that the latter components adversely affect crystallization, thus making direct production of high quality white sugar impossible.

Addition of a membrane filtration step to the juice purification process, which has been intensively explored over the last decade helps eliminate some of the large molecular weight components of cane juice. It has been proven that micro- or ultrafiltration is capable of providing a drastic improvement in the quality of crystallized sugar. However, depending on pore size membrane filtration usually removes components with molecular weights exceeding 50,000-1,000,000. Therefore, it does not change the content of inorganic salts, invert sugars or lower molecular weight colorants present in juice. This is the reason that most researchers looking for direct production of white sugar in the cane mills have concentrated their efforts in finding a process that could complement membrane filtration as well as eliminate the remaining low molecular weight non-sugars. As a result various adsorptive and membrane decolorization and demineralization methods have been tested with limited success. Usually a method powerful for removal of e.g. colorants, such as decolorization resins, fails to reduce ash or invert components of juice to acceptable levels.

Modern technology offers a variety of unit operations, combinations of which allows one to eventually obtain sugar crystals satisfying the requirements for refined sugar. The search for economically feasible processes of white sugar production in the cane mills should be the main goal of today's process development. Evidently, the feasibility of new technologies will depend on

market goals and conditions, product specifications, size of a factory, length of campaign and other factors.

In an effort to develop a new technology eliminating the need of lime for purification of beet juice Amalgamated Research Inc. has studied the application of continuous simulated moving bed (SMB) chromatography. Our previous experience with cane molasses desugarization has indicated that cane non-sugars could be efficiently removed from the feed solutions. Since the mechanism of purification is based on size and ion exclusion, chromatography appears to be especially powerful in eliminating inorganic salts and large molecules, such as dextrans, colorants, starch, etc. Although separation of an invert stream from conventional SMB was technically possible, the degree of elimination had not exceeded 30-40%. With the introduction of ARi's new coupled loop chromatographic process much higher invert elimination may be achieved. The process tested therefore was ARi's beet/cane raw juice purification process with chromatography step using coupled loop method.

Clarified cane juice was filtered through an ultrafiltration membrane with molecular weight cut-off of about 100,000 Dalton and softened using Rohm & Haas IMAC HP1110 strong cation exchange resin. The resulting juice was then evaporated to about 70% DS and shipped from a cane mill to ARi at Twin Falls, where the following tests were performed

Typically each chromatographic test continued for at least 200-300 hours, which was sufficient to establish the equilibrated product concentrations and make several changes to optimize the separator performance. Table 1 contains the analytical data of representative samples of softened, ultrafiltered and concentrated clarified juice and final chromatographic extracts. Color of feed material for chromatographic trials was about 40-50% higher in comparison to typical samples of meladura. Because of the nature of the coupled loop process the feed and extract samples are not taken at the same time. The samples show comparison of the initial feed material into Loop 1 of the process and the final extract out of Loop 2.

Material balances were calculated for each test. Data presented in Table 2 show average percent elimination of components from the feed material. Sugar recovery in the chromatographic process reaches 93-94%.

The sugar-enriched fraction (extract) was concentrated in a continuous pilot evaporator and then crystallized in a batch 60-liter vacuum pan. Sugar crystals were separated in a pilot Boch centrifuge. To verify the performance of the vacuum pan and centrifuge samples of thick juice from the Twin Falls beet sugar plant were crystallized. Quality of sugar obtained in the pilot trials was compared with the factory samples. Although the crystal yield and size were similar to the factory samples, it was difficult to reproduce the washing regime in a pilot centrifuge. Because the pilot tests usually yielded sugar crystals with higher color, it was concluded that production of white sugar in the pilot pan would give reliable and conservative information about the process capability.

Several batches of concentrated extract were crystallized. Although color of feed material ranged from 1800 to 3800 RBU, color of crystallized sugar consistently did not exceed 22-23 RBU after standard washing procedure. The invert content of final sugar samples was 0.0067% (based on D.S.), ash content- less than 0.001%. It is noted that the use of traditional color measurement for

estimates of different intermediate products, such as membrane permeates or chromatographic extract is meaningful only if samples from the same process are compared. Quality of final sugar is the only reliable way to compare the various alternatives.

For comparison purposes we included a third sample, when sugar was boiled from a sample of concentrated extract obtained from softened factory meladura filtered using the conventional (non-membrane) method. Since this sample was not prepared and shipped with proper caution, additional handling and evaporation were required, which resulted in extract with very high color (about 10,000 RBU). However, the sugar crystals had relatively low color and extremely low ash content, 50 RBU and 0.001%, respectively. Obviously, chromatography has more pronounced effect on sugar quality than membrane filtration.

It is evident from crystallization results that sugar crystallized from chromatographic extracts meets the requirements for refined sugar. A combination of membrane filtration, softening and chromatography applied to clarified cane allows for a large portion of non-sugars to be eliminated. Moreover, since the new process removes most of the high molecular weight components, crystallization of white sugar is possible from syrups with relatively high color. Remaining colorants are easily removed from the sugar crystals using the standard washing procedures. The test program will continue during the campaign of 1998-99.

Table 1
Analysis of feed material and final extracts for chromatography tests

Sample No.	RDS	AP	GC Purity	pH	ICUMSA Color	Invert	g/100 g DS			
							Na	K	Ca	Mg
Feed 1	68.5	87.2	86.5	8.7	16,600	2.85	0.058	2.72	0.001	0.001
Feed 2	67.9	86.4	86.2	9.2	18,000	2.23	0.030	3.01	0.001	0.001
Extract 1	36.1	98.1	97.5	9.0	2,200	0.69	0.005	0.10	0.000	0.000
Extract 2	38.6	97.5	97.0	8.4	1,300	0.51	0.014	0.35	0.000	0.000

Table 2
Percent elimination of various non-sugars in the pilot chromatographic process

Component	Total non-sugars	Invert	Color	Na	K	Ca	Mg
Percent elimination	85-86	60-80	90-92	97-98	97-98	92-93	98-99