

CHROMATOGRAPHIC DESUGARIZATION OF SYRUPS IN CANE MILLS

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Abstract

This paper reviews recent research work and developments in the “new separation technologies” of membrane filtration, juice softening and chromatographic separations. Their applicability in the beet and cane sugar industries is discussed, particularly for sugar quality (especially decolorization), purity enhancement and sugar recovery.

Research results are reported, leading to conclusions regarding current limitations and future potential of the processes. Directions for future development are discussed.

Review of new separation technologies

In this paper the term “new separation technologies” will be applied to the concepts being proposed for significant improvement of either recovery or sugar quality in the cane industry. Most new technologies comprise the unit operations using ion exchange and chromatographic resins and/or membrane filtration processes. These processes have been attracting the attention of sugar researchers for the last few decades. The advances in polymer science and membrane manufacture have spurred research and development efforts worldwide. Below we have attempted summarizing major trends in new process development during the last decade with the emphasis on membrane and chromatographic technologies. Our latest results on chromatographic desugarization of syrups in cane mills are presented and discussed.

The main impetus behind the use of membrane technology in the cane sugar industry is the improvement of quality and recovery of raw sugar. It is interesting that the first efforts to use ultrafiltration membranes for cane juices and syrups were made in the 1970's. W. K. Nielsen and his colleagues (1982) gave an extensive review and summary of these efforts. Specific features of sugar applications created challenges for the membrane industry. Despite intensive pilot testing no large membrane installations are in use in the industry to date. Information on a large-scale plant for ultrafiltration of clarified cane juice with ceramic membranes was reported in a SPRI meeting (Theoleyre 1996). Two years after the startup, the installation reportedly was still in the development phase with estimated return on investment of about 8% (Kwok, 1996). Several other membrane manufacturers are currently researching the idea of using MF/UF membranes in the sugar mills and refineries. The latest reports by Koch Membrane Systems and Graver Technologies on potential application of polymeric spiral wound and stainless steel membranes, respectively, for cane juices and syrups are very encouraging (Eringis, 1999; Wittwer, 1999). Over the last few years membrane quality has been drastically improved and

large-scale pilot systems have been operated worldwide. Test results on several different MF/UF membranes were recently reported by Alvarez (1999). Obviously, objective information of this sort is needed to clarify the benefits of membrane technology for cane mills or refineries. Since MF and UF membranes typically provide very little non-sugar removal, an additional purification process may be required. Several processes have been proposed to meet this objective.

Monclin (1996) has suggested complementing micro/ultrafiltration of mixed or clarified cane juice by adsorptive decolorization. Although the process is capable of removing high molecular weight materials and certain colorants, it does not reduce the amount of monovalent cations and invert sugars in the solution, which comprise a major portion of non-sucroses in the juice stream. An alternative approach where UF was followed by nanofiltration has been proposed by Saska (1995). In both cases the authors claim that white sugar can be produced directly in cane mills. Microfiltration or ultrafiltration may also be used as a pretreatment prior to chromatographic separation according to the process developed and patented by Amalgamated Research Inc. (Kearney *et al.*, 1995). The results show that very high quality white sugar can be manufactured as a result of this process (Kochergin *et al.*, 1999). After successful implementation of molasses desugarization in the beet industry efforts have been made to use ion exclusion chromatography on low-purity cane syrups. Recently Peacock *et al.* (1999) reported that the processes are not yet economically feasible mainly due to the pretreatment difficulties.

It should be noted that much information on new process performance has not addressed the issues critical for proper assessment of developed technologies. It is critical to realize the fundamentals of various separation processes and their effect on components of technical sugar solutions.

Membrane performance: Expectations and reality

Although membrane separation characteristics strongly depend on the average pore size, the mechanism of separation is much more complicated than simple “screening”. Formation of a dynamic layer on the surface, concentration of dissolved and suspended solids, and the presence of surfactants are among the factors critical for membrane performance. According to traditional classification, membranes are rated by pore size in the microfiltration range and by “molecular weight cut-off” (MWCO) in ultrafiltration.

Analysis of the composition of various sugar juices and syrups is very helpful in evaluating expected membrane performance. Although only experimental results can provide the true infor-

mation on membrane performance, preliminary analysis may save time and resources for a test program and provide information on the nature of potential foulants.

Since we have not found a detailed analysis of a sample of clarified cane juice in the literature, we have analyzed a sample of beet diffusion juice from a factory in southern Idaho (USA). The data is shown in Table 1. Chemical composition of cane clarified juice differs significantly from beet diffusion juice, however, the distribution of the components by molecular weight is very similar. A simple comparison of molecular size of juice constituents with pore size of membranes indicates that most high molecular weight components (molecular weight exceeding several hundred thousand units) can be removed by

microfiltration or “loose” ultrafiltration membranes. Therefore, the claims of some researchers that UF membranes have much more pronounced effect on sugar quality than the MF membranes need to be thoroughly evaluated.

Based on our simple preliminary analysis of juice properties it may be concluded that the use of MF/UF membranes may be feasible for the removal of suspended solids, colloidal material and other high molecular weight compounds, but they perform poorly for separation of most of the dissolved non-sugar components.

Analysis of data in Tables 2 and 3 can give an answer to the long-debated question what purity rise can be expected across

Table 1. Typical Analysis of Beet Diffusion Juice (Southern Idaho).

Component	Concentration		Molecular weight
	% on DS	% on non-sugars	
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 non-sugars	1.26	10.50	--
Total non-sucrose	12.00	100.00	--
Total solids	100.00		--

* Concentration of glutamine and amino acids is calculated based on molasses content of about 9% on non-sugars.

** Information was not available.

*** Calcium and magnesium are calculated based on hardness level of 12 meq/100 DS.

the MF/UF membranes. Table 2 contains calculated numbers of purity increase corresponding to different levels of non-sugar removal for an 88 purity juice sample. Under the assumption that one half of the unaccounted non-sugars in Table 1 is rejected by UF membranes, maximal theoretical non-sugar elimination can not exceed approximately 10%. The purity difference between the feed juice and permeate increases with decrease in feed juice purity assuming the same level of non-sugar elimination. Expected values of purity rise across a membrane at 10% non-sugar elimination are listed in Table 3. Therefore, the expected purity change across a MF or UF membrane should not exceed more than one purity point (may be slightly higher for low purity juices).

Table 2. Juice purity increase as a function of non-sugar elimination.

Juice purity 88 Non-sugars, % DS 12		
Non-Sugar Elimination, %	Permeate Purity, %	Purity Increase, Points
5	88.53	0.53
10	89.07	1.07
15	89.61	1.61
20	90.16	2.16
25	90.72	2.72
30	91.29	3.29
35	91.86	3.86
40	92.44	4.44
45	93.02	5.02

It is also important to analyze the influence of membrane filtration on other parameters critical for various processing steps. In our previous paper (Kočergerin, 1998) we have summarized some information on removal of color and dextrans from various technical sugar solutions.

Review of cane molasses desugarization process

Desugarization of molasses by chromatography has proven to be a profitable operation in the beet sugar industry. Because of the favorable ratio between the prices of white sugar and molasses most companies in the USA as well as several companies in Europe and Asia have built large chromatographic desugarization plants. The daily capacity of the largest plants exceeds 600 tons of molasses per day. Apparent purity of final beet molasses typically fluctuates between 58 and 62%. With the latest chromatographic technologies capable of extracting

over 99% of the sugar at an extract purity exceeding 96%, molasses desugarization attracts more attention from sugar technologists worldwide. After extract crystallization, 86-88% of the sugar can be sold as product. Overall beet factory recovery with molasses desugarization exceeds 92%.

Despite continuous interest from cane sugar technologists the cane industry has been traditionally lagging behind in the application of chromatographic desugarization. We believe it is important to analyze the reasons and evaluate if the technologies are available to incorporate chromatographic desugarization into the cane sugar industry.

Although the initial raw (clarified) juice purities are similar to beet raw juice (about 86-88%), crystallization in cane mills results in a molasses apparent purity of about 28-35%. The reason for the low purity of cane molasses is the higher concentration of invert sugar, which is known to have a negative melassigenic effect. At 30% apparent purity C-molasses contains about one-half of the sugar comparing to an equivalent sample of 60 purity beet molasses. It should be emphasized, however, that the use of apparent purity measurements can lead to underestimation of sugar content in cane molasses (the true purity is typically several points higher). Generally lower sugar content in cane molasses and higher overall extraction compared to beet factories are among the reasons why chromatographic technology appears to be less feasible in the cane industry.

To understand another important reason one must analyze the requirements of chromatographic desugarization. The process is carried out in large columns (diameters up to 7 meters) filled with ion-exchange resin in a monovalent cation form. Resin particle size typically varies between 300 and 390 micron. To achieve good sugar separation the feed material should be practically free of divalent cations such as calcium and magnesium. Feed material should also be free of any suspended solids to prevent plugging of the resin bed. Clearly a filtration step should precede molasses softening and desugarization operation.

Currently the lack of a practical and effective technology capable of suspended solids removal from cane molasses reduces the feasibility of chromatographic desugarization. The concentration and make-up of the suspended solids in cane B- and C-molasses ranges greatly depending on the growing area. However, most samples contain from as low as 0.5 to as high as 7-8% by volume suspended solids. This factor alone can result in large filtration losses. Washing of filtration sludge to reduce the sugar loss results in additional evaporation cost. To make things worse, the suspended particle size distribution is extremely wide ranging from relatively large particles of a few hundred μm down to particles of a fraction of a μm . The particle size distribution of some samples analyzed at Amalgamated Research Inc. (ARi)

Table 3. Expected purity increase at 10 % non-sugar removal.

Juice Purity	82	83	84	85	86	87	88	89	90
Purity rise @ 10% NS elimination	1.50	1.44	1.37	1.29	1.22	1.15	1.07	0.99	0.91

shows that as much as 50% of suspended particles are smaller than 2 μm in size.

It is interesting to note that beet molasses is typically filtered prior to desugarization using conventional pressure filtration with precoat and body feeding. Diatomaceous earth or perlites are used for this purpose with average particle sizes of 5-10 μm . The resulting filtrate does not contain significant suspended solids. The concentration of suspended solids in beet molasses is typically quite low (several hundred ppm). The same filtration method applied to cane molasses does not produce filtrate completely free of suspended solids. Extra fine particles penetrate through filtration media and may potentially accumulate in the chromatographic columns raising the pressure drop and causing system shutdown. Also, because of the large volume of suspended solids, filtration rates are extremely low. To reduce the suspended solids content a centrifugation step has been added before filtration in some plants.

The alternative suspended solids removal methods, such as micro- or ultrafiltration, have been tested on cane molasses without much success. Although membrane filters produce permeate of exceptional quality, i.e. absolutely free of suspended solids, low expected fluxes and fouling characteristics of cane molasses do not make membrane filtration look practical at present.

The above discussion leaves very little doubt that the use of molasses desugarization in the cane industry appears impractical. We believe, however, that the cane sugar industry can benefit from the chromatographic separation technique applied to process streams other than molasses. The new technology development providing benefits of additional recovery and higher quality raw sugar is discussed below.

Pilot equipment and procedures

The idea of chromatographic separation of high purity juice has been originally developed for beet raw juice (Kearney, 1996). Later, the concept was extended for application in the cane industry. The process includes the steps of membrane filtration with the purpose of suspended solids removal, softening and evaporation. The syrup then is fed into chromatographic columns where most of the non-sugars are eliminated. The sugar-rich fraction extract then is concentrated and crystallized to yield white sugar. The sugar-lean fraction raffinate, may be used as cattle feed. It is important to understand that the size of a chromatographic system is based mainly on the non-sugar loading and, with a given non-sugar load, is the same for clarified juice and molasses.

The concept of white sugar production directly in a cane mill was proven in the first year of our study (Kochergin *et al.*, 1999). Samples of sugar have been obtained satisfying all requirements of white sugar. The objectives of the current study were optimizing of chromatographic process characteristics and obtaining reliable information required for a feasibility study. Therefore, the size of pilot installations was chosen to ensure the reliable transfer to industrial scale.

Experimental work was carried out for the last two crop seasons. Pilot membrane filtration, softening and evaporator in-

stallations were set up at Sugar Cane Growers Cooperative of Florida in Belle Glade, FL. Evaporated syrup was shipped to ARI in Twin Falls, ID where chromatographic and crystallization studies were performed.

A pilot membrane installation with Koch spiral wound ultrafiltration membranes (molecular weight cut-off about 80,000 Dalton) processed 22 m^3/hour of clarified juice. Juice at 12-14% DS was pre-filtered through a 100-micron wedge-wire screen to remove fibrous material that could potentially plug the feed channels. The membrane installation has been in operation continuously 24 hours per day, several days a week with periodic interruptions for cleaning. Since the pilot setup was comprised of commercial size modules the results are completely representative of an industrial installation.

A portion of permeate stream was passed through a 30 cm diameter pilot softener column filled with Rohm & Haas IMAC HP1110 strong cation exchange resin. Although the feed material contained more than 22 meq/100 DS of calcium and magnesium ions, the effluent was free of divalent cations. The softened juice has been fed into a fully automated continuous forced circulation vacuum-evaporator (capacity about 1.5 l/min based on water removed, temperature 80-85°C). The resulting syrup concentrated to about 70% DS was packed in the 0.8 m^3 containers and shipped to the ARI facility. Thus the samples of syrup were composites of several days of operation.

The simulated moving bed (SMB) chromatographic pilot plant in Twin Falls is completely automated and is operated continuously for 200-300 hours for each test. Because of the nature of the SMB process, it is necessary to operate the plant for at least 12-24 hours before the system reaches complete equilibrium. Optimization of separator performance may take several changes during each test. Three tests with syrup samples representing various periods of grinding season have been performed. We have used the recently developed method of Coupled Loop Chromatography that achieved very high sugar recovery along with elimination of most invert sugars (Kochergin and Kearney, 1997).

Crystallization studies have been carried out in an automated 60-liter batch vacuum pan. Crystals have been separated in a pilot centrifuge (bowl diameter 20 cm). To verify the performance of both pan and centrifuge, samples of thick juice from the Twin Falls beet sugar factory were crystallized. The quality of sugar obtained in the pilot trials was compared with the factory samples. Although the crystal yield and size were very similar to the factory samples, it was impossible to reproduce the washing regime of the industrial centrifuges on a small scale. Since the sugar samples from the pilot centrifuge had consistently higher color than the factory samples, it was concluded that the pilot results would give conservative information about the process.

Crystallization experiments have been carried out using previous experience with seed quantity, temperature and feed rates for high purity extracts. The supersaturation level of 1.25 has been selected for 97-98 AP syrups. Fondant used for seeding in the beet sugar plant has been applied. The crystallization temperature was maintained at 73-73.5°C. Initially 17-18 liters of feed were brought into the pan and the operating tempera-

ture was established. The seed was introduced and held for about 15 minutes, and after that the feeding was continued for 80-120 minutes. Afterwards the massecuite was concentrated for another 30 minutes and then centrifuged and washed. The sugar samples were placed in a pilot dryer for 20 minutes and then analyzed. A pilot dryer was used simply to minimize caking of the samples.

Results and Discussion

Chromatography

Performance of the chromatographic pilot plant was significantly improved in comparison with previously reported data. Chromatographic separation results are summarized in Tables 4 and 5. Table 4 contains the analytical data for representative samples of feed material (softened, ultrafiltered and concentrated clarified juice), final chromatographic extracts and invert fraction. Because of the nature of the Coupled Loop process the feed and extract samples were 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. The Coupled Loop process can generate the third stream containing mostly invert sugars. This stream can be either used separately or combined with the raffinate. Material balances were calculated for each test. Data presented in Table 5 show the average percent elimination of components from feed material to a chromatographic separator. Sugar recovery in the chromatographic process exceeds 99% (based on feed syrup). Calculation of material balances has shown that recovery of sugar in the mill can be improved by 6-7 points (for example, recovery excluding bagasse losses is 89%, expected recovery is over 96%). It corresponds to approximately 8% increase in sugar production, assuming final molasses purity of 60%. The recovery can be greater with lower molasses purity. This possibility has yet to be proven.

Crystallization Studies

The results of crystallization of four extract samples are summarized in Table 6. Different wash times have been applied. The washing procedure was manual, the quantity of wash water was measured and calculated based on fillmass volume. It is important to note that manual washing is no match to washing procedures in industrial centrifuges. Sample X-1 clearly indicates that the increased amount of wash water results in better quality sugar. This is not the case for the syrups obtained using different technology. The results show that very

Table 5. Percent elimination of non-sugars in the pilot chromatographic tests

Component	Total NS	Invert	Color	Na	K
Percent Elimination	over 90	85-90	90-92	97-98	97-98

high quality white sugar can be crystallized. All four samples exceeded specifications for white sugar in all aspects.

The quality of raw sugar obtained using the new technology would be significantly better compared to conventional technology. The unwashed samples of crystallized extract are representative of the quality of the "new" raw sugar. Data in Table 7 illustrate the differences between conventional raw sugar samples and unwashed samples obtained from extract. The raw sugar samples were collected at different days of Belle Glade mill operation. It is important to note that a small amount of wash water has been used in the mill. During crystallization studies the purity of unwashed sugar was not routinely analyzed. However, several samples showed that the apparent purity exceeded 99.5%. The purity of conventional raw sugar samples fluctuated between 98.5 and 99%.

The results demonstrate the superior quality of raw sugars that can be obtained from extracts. Also the data correlate well with the performance of chromatographic separation. Membrane filtration is obviously responsible for reduced turbidity and partial color reduction. The low numbers for invert and ash are due to the contribution of the chromatographic process that provides very high elimination of these components (see Table 5). Conversion from raw to white sugar can be achieved by simple washing in the centrifuges. Such process may be useful for companies operating their own refineries.

To evaluate the relative contributions of membrane filtration and chromatography in the improvement of sugar quality we have crystallized syrup made from softened and ultrafiltered clarified juice without chromatography. Small batches of massecuite (about 800 grams each) were washed consecutively with different amounts of hot water. Sugar color was measured for each batch. The results plotted in Figure 1 demonstrate the effect of washing on color transfer to sugar crystals. For illustrative purpose the inverse values of color transfer ratio (which is defined as ratio of colors of crystal to feed syrup) are plotted along the Y-axis. A similar graph has been plotted for samples of sugar obtained from chromatographic extracts. The graphs clearly indicate that the addition of the chromatographic process to membrane filtration results in much better sugar quality.

Table 4. Analysis of three fractions from chromatographic tests.

Sample	RDS	A.P.	G.C.	pH	Color	Invert	Na	K
Number	%	%	purity, %		ICUMSA	gms/100 RDS		
Feed 1	66.3	87.2	89.3	8.9	11,056	2.76	1.207	0.352
Feed 2	66.3	87.6	89.7	8.7	10,708	2.77	1.164	0.356
Invert 1	5.7	-80.7	3.4	6.0	9,883	78.21	1.009	0.567
Invert 2	7.1	-62.4	3.5	5.8	7,489	65.28	0.803	0.356
Extract 1	40.9	97.6	97.9	9.1	1,337	-	0.180	0.430
Extract 2	40.9	98.1	98.4	8.9	860	0.31	0.150	0.330

In the case of chromatographic pretreatment of syrup, color can be easily washed off the surface of a sugar crystal. On the other hand, in case of membrane filtration, incremental addition of wash water does not provide much color decrease. The

latter is an indication that the colorants are occluded in the crystal.

An interesting study by L. Bento (1997) may provide an explanation for the observed phenomenon. In the experiment a gel-

Table 6. Crystallization Results.

	% Wash Water	RDS	A.P.	Color	Turbidity	MA	CV	Cond. Ash	Invert
	/fillmass			RBU	(at 720 nm)			%	g/100 DS
Pan X-1									
Feed		69.53	97.78	2322	386	–	–		0.27
Fillmass		91.07	97.38	2215	274	–	–		0.23
True Green		77.92	93.18	6834	789	–	–		0.58
Green		75.60	94.77	4271	559	–	–		0.44
Unwashed sugar				641	37			0.165	
Washed sugar 1	4.2			31	24	425	38		0.01
Washed sugar 2	5.6			7.8	32	372	44	0.005	0.01
Pan X-2									
Feed		70.22	97.59	2491	192	–	–		0.26
Fillmass		90.17	97.21	2515	256	–	–		0.22
True Green		78.72	94.45	6203	733	–	–		0.44
Green		75.95	95.13	4074	607	–	–		0.38
Unwashed sugar				501	10	440	35	0.108	0.03
Washed sugar 1	3.1			23.7	14	380	39	0.006	0.01
Pan X-3									
Feed		67.68	97.78	1875	256	–	–		0.23
Fillmass		91.34	97.57	2315	253	–	–		0.18
True Green		80.86	94.76	6726	737	–	–		0.41
Green		77.82	95.58	4940	617	–	–		0.35
Unwashed sugar				642	18	504	28	0.144	0.03
Washed sugar 1	4.2			24.1	10	415	36	0.005	0.11
Pan X-6									
Feed		69.87	97.34	2448	83	–	–		0.26
Fillmass		91.26	97.34	2864	295	–	–		0.23
True Green		79.78	93.44	7490	771	–	–		0.52
Green		77.82	95.16	5359	543	–	–		0.39
Unwashed sugar				623	15	527	27	0.136	0.03
Washed sugar 1	3.8			32	8	443	35	0.006	0.01

Table 7. Comparison of Quality of Raw Sugar obtained from Chromatographic Extract and Conventional Raws.

Pan #		Extract Pans*				Conventional Raw**		
		X-1	X-2	X-3	X-4	Day 7	Day 17	Day 37
Color (RBU)	Syrup	2322	2491	1875	2448	1802	1226	1056
	Unwashed sugar	641	501	642	623			
Invert (g/100 DS)	Syrup	0.27	0.25	0.23	0.27	0.71	0.47	0.51
	Unwashed sugar	–	0.028	0.03	0.03			
Conductivity Ash, %	Unwashed sugar	0.165	0.108	0.144	0.136	0.40	0.27	0.24
Turbidity (at 720 nm)	Unwashed sugar	37	10	18	15	1030	289	281

* No wash water applied

** Small amount of wash water is used in conventional centrifuges

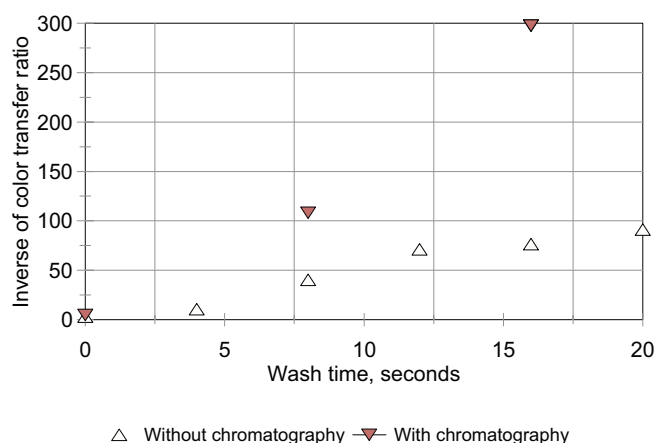


Figure 1. Effect of washing on sugar quality.

permeation chromatography method was used to study the distribution of colorants by their molecular weight. He has isolated three fractions of sugar colorants within the range from 2500 to several million Dalton in refinery streams and observed the distribution of those compounds between sugar and various syrups. It has been confirmed that the high MW compounds tend to stay with both unwashed and washed sugar. It is interesting that the compounds with MW ranging between 2,5 and 12 kD also show weaker but similar tendency. That explains the limited effect of micro- and ultrafiltration on color elimination from the final product observed in our tests.

The low MW colorants can be removed more efficiently by chromatographic methods because of significant MW and charge differences of colorants and sucrose. In addition chromatographic methods are very efficient in removal of ash and invert sugars. Because of the different principle from the membrane filtration separation mechanism, chromatography is much more selective in the removal of color molecules, dextrans, starches, etc. Separation of substances with small difference

in molecular weight (that becomes a major obstacle for membrane separations) is easily accomplished with simulated moving bed (SMB) chromatography.

Recently we attempted analyzing the effect of new technologies on the quality of sugar (Kochergin, 1999). The data was compiled on color transfer ratios for different processes in both the beet and cane sugar industries. Although the values presented in Table 8 are approximate, they clearly show that in the absence of any filtration, such as in most cane mills, the color transfer ratio is maximal. Low values of color transfer ratio correspond to better purification methods. The ratio decreases for the refinery syrups where filtration and purification is added. Use of ultrafiltration results in better quality sugar, although the effect on color removal is not very significant. The color transfer ratio for a combination of ultrafiltration and chromatography is quite low. Interestingly, the color removal from cane juice and syrups becomes comparable with beet syrups treated in a similar manner.

Conclusions

1. The introduction of new separation technologies dramatically affects the quality of final product. More objective information on large-scale pilots performance is needed to evaluate the feasibility of new technologies.
2. The technology using chromatography for purification of softened and ultrafiltered clarified juice can provide about 8 % increase in sugar production in a cane mill. Depending on the preference, white sugar or very high quality raw sugar can be produced.
3. The chromatographic Coupled Loop process provides invert, color, and ash reduction sufficient for direct production of white sugar from relatively high color syrups. Understanding that relatively high color syrups may yield low

Table 8. Relative color of syrups and sugar in various process.

Process	Syrup pretreatment	Syrup color, ICUMSA	First strike sugar color ICUMSA	Color transfer ratio *100
Cane mill (conventional)	Coagulation, Settling	15,000	3,000(unwashed)	20.0
Cane refinery	Affination , filtration, decolorization	300	10(washed)	3.3
UF and NF (Saska, 1995)	UF/NF	3590	304 (unwashed) 46 (washed)	8.5 1.3
Ultrafiltered cane clarified juice (Kwok, 1996)	Ultrafiltration	15000	600	4.0
Beet process(conventional)	Chemical treatment, DE filtration	3,000	30(washed)	1.0
Chromatographic extract from beet molasses	Conventional and chromatography	6,000	30(washed)	0.5
Chromatographic extract from cane clarified juice (new process)	Ultrafiltration and chromatography	4500	30(washed)	0.7

color sugar will lead to some interesting process developments previously neglected or assumed impractical.

4. Regardless of color reduction in membrane filtration steps the effect on the final product may be more significant. Membrane filtration indirectly influences the ash and invert content of the final sugar, because of the removal of high MW components that adversely affect the crystallization process. Since membranes do not remove certain impurities, another process is required to accomplish the juice purification sufficient to produce white sugar. Because of a negligible purity rise across the membranes, no recovery benefits can be realized.
5. Estimating color values of the intermediate products may give erroneous information about process efficiency. Color values in various processing schemes represent a completely different combination of colorants. It is critical to understand which portion of the molecular weight distribution range a certain separation process is affecting.
6. Cane molasses desugarization is not practical for use in the cane mills because of the difficulties in pretreatment. Even if membrane filtration were applied on clarified juice, the resulting molasses would still contain suspended solids. This will require a second filtration step prior to chromatography on molasses.

REFERENCES

- Alvarez, J (1999). Ultra-filtration technology applications in the raw sugar industry. *S.P.R.I. Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, September 8-10*: in press.
- Bento, L, *et al.* (1997). Improved analysis of sugar colorants. *Int Sug J*. Vol. 99, No. 1187: 555-562.
- Kearney, M, Kochergin, V, Petersen, K and Velasquez, L. (1995). Sugar juice purification process. *U.S. Patent 5,466,294* (1995), *Re# 36361* (1999).
- Kearney, M and Eugene Rearick, D. (1996). Raw juice chromatographic separation process. *Int Sug J*, Vol. 98, No. 1168B: 144-148.
- Kochergin, V. (1998). The future of membrane technology in the sugar industry: importance of proper testing. *Proc S.P.R.I. Conference, March 22-25, 1998, Savannah, GA*: 40-56.
- Kochergin, V, Kearney, M, *et al.* (1999). *Proc Int Soc Sug Cane Technol XXIII (Preprint vol)*: 374-377.
- Kochergin, V and Kearney, M. (1997). Multicomponent separation using simulated moving bed chromatography. *Proc. Am Inst Ch Eng annual meeting, Los Angeles, CA, Nov. 16-21*, vol. 2: 1539-1544.
- Kochergin, V, *et al.* (1999). New separation technologies: Effect on sugar quality. *S.P.R.I. Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, September 8-10*: in press.
- Kwok, R. (1996). Ultrafiltration/softening of clarified cane juice. *Proc S.P.R.I. workshop on separation process in sugar industry, ed. M. Clarke*: 87-99.
- McArdle, J and Eringis, A. (1999) Performance of high temperature polymeric spiral membranes in the cane and beet sugar industries. *S.P.R.I. Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, September 8-10*: in press.
- Monclin, JP and Willett, SC. (1996). The A.B.C. process for the direct production of refined sugar from cane juice. *Proc S.P.R.I. workshop on separation process in sugar industry, ed. M. Clarke*: 16-28.
- Nielsen, WK, Kristensen, S and Madsen, RF. (1982). Prospects and possibilities of membrane filtration systems within the beet and cane industry. *Sugar Technology Reviews*, 9: 59-117.
- Peacock, S. (1999). Invert from cane molasses using chromatographic techniques. *S.P.R.I. Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, September 8-10*: in press.
- Saska, M, *et al.* (1995). Direct production of white cane sugar with clarification and decolorization membranes. *Sugar Journal, Nov. 1995*: 19-21.
- Theoleyre, MA. (1996). Membrane technology in the sugar industry. *Proc S.P.R.I. workshop on separation process in sugar industry, ed. M. Clarke*: 55-69.
- Wittwer S. (1999). Applications for stainless steel crossflow membranes in sugar processing. *S.P.R.I. Symposium on Advanced Technology for Raw Sugar and Cane and Beet Refined Sugar Production, September 8-10*: in press.