MEMBRANE FILTRATION OF VARIOUS SUGAR SOLUTIONS

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INTRODUCTION

There are several important reasons for recent breakthrough of membrane technologies into most of the major industries. Drastic improvement of membrane materials has been made during the last 10-15 years. New environmental regulations impose additional requirements on conventional filtration processes resulting in increased cost of filter cake handling and disposal. Lower costs and an extended lifetime of the membranes are among the factors to be mentioned in this context. These and other factors make membrane technology applicable in the areas where it was not previously regarded feasible. Therefore, the time has come to reconsider replacement of conventional filtration and to develop the new applications for membrane separations.

The sugar industry is one of the few areas that membrane “explosion” has not reached. It is even more surprising taking into account that first research papers on membrane applications in sugar industry appeared in the early 1970’s. R.F. Madsen1 reported testing UF cellulose acetate membranes for beet sugar juices’ purification and RO membranes for juice concentration. An idea of replacing conventional juice purification by membrane process fascinated sugar technologists for many years. Numerous efforts have been done to test the feasibility of the new processes2,3,4. W. K. Nielsen and his colleagues gave an extensive review and summary of these efforts5. Variations in juice properties and analytical procedures shown in the papers of different authors make it difficult to conclude whether or not membranes can replace traditional processes. Information is available in the literature6 on use of membranes for clarification and decolorization of cane syrups.

Only recently a large-scale system was installed for ultrafiltration of clarified cane juice. The system utilizes Kerasep ceramic membranes. Details on system parameters and operation can be found in the literature7,8.

The purpose of this paper is to review possible applications for membrane technology in the sugar industry based on the analysis of sugar processing technology and recent experimental results. Applications where micro- or ultrafiltration processes are used as a pretreatment of feed to chromatographic separation will be discussed.

Potential applications for membrane technology

Most of the applications in the sugar industry would impose specific requirements on membrane characteristics:

1. High fluxes (a typical sugar plant produces 1000-2000 gpm of juice).
2. Temperatures above 85°C to prevent bacterial growth.

3. Presence of small quantities of abrasive materials in the raw juice streams.

4. High concentration factors required to minimize sugar losses.

Possible applications for membrane technology in beet and cane sugar industry can be conditionally divided into two groups: replacement of conventional filtration processes and the new developments. Each potential membrane process will need to go through a development stage and subsequent economic evaluation. Replacement of traditional filtration is still difficult to justify but the new projects may become more feasible with the increased requirements on handling of waste filter-aid. Particularly, cost of the equipment necessary to dewater filter-aid sludge in some applications may be comparable to the cost of a membrane system. This will make membrane technology look more attractive to sugar technologists.

**Group 1**

1. Standard liquor filtration (some companies apply two-stage conventional filtration). This process will eliminate filter-aid usage and disposal but still requires appropriate concentrate handling process to reduce sugar loss.

2. Some factories store thick juice during slicing campaign and process it later in the season. Thick juice is normally filtered prior to storage to reduce bacterial counts. Juice sterilization by microfiltration can significantly reduce storage losses.

3. Molasses desugarization systems in the beet industry are very sensitive to the presence of suspended solids in the feed streams. Micro- or ultrafiltration can be extremely efficient for this application.

**Group 2**

1. Raw juice microfiltration or ultrafiltration as a part of pretreatment prior to chromatographic separation according to the process patented by The Amalgamated Sugar Company. Pretreated and filtered raw juice is softened and evaporated. Resulting syrup is purified using chromatographic separation. The process has been successfully piloted for four years. Details about the process can be found in the paper by M. Kearney and D. E. Rearick.

2. The same approach like the one referenced in paragraph one can be applied to cane juice purification. It is proven that chromatography is capable of removal cane non-sugars and color very efficiently. Membrane pretreatment will be necessary prior to feeding juice to a resin bed.

3. Raw juice ultrafiltration as a replacement to conventional purification method is still difficult to justify as a stand-alone process. Permeate is more likely to be post-treated with lime to achieve a purity increase comparable to liming and carbonation.
4. Microfiltered raw juice can be stored and processed later. After appropriate testing the process may be useful for factories with lime kilns undersized to handle an increased slice rate.

5. Cane molasses pretreatment prior to chromatography should be developed on case to case basis due to tremendous differences in molasses properties from various sources.

6. Micro- or ultrafiltration may be applied for both press water sterilization and suspended solids removal. Sterilization will not require heating and subsequent cooling. This process can be justified if reduction of bacteria counts and associated unaccounted loss could be accomplished.

7. Microfiltration of mixed cane juice followed by adsorption will remove high molecular weight materials. Feasibility of this process should be carefully studied since adsorption does not reduce amount of highly melassigenic monovalent cations in the solution which comprise a major portion of non-sugars in the juice stream.

8. Cane juice color may be reduced by the UF in the cane mills and refineries.

New ideas on membrane applications continue to appear on a daily basis when new information on membrane performance becomes available. Setting priorities overall is difficult due to the differences in companies’ strategic planning. Priorities are significantly affected by new EPA regulations, cost of power and membrane systems.

**Raw juice purification using simulated moving bed chromatography**

Chromatographic separation of raw beet or cane juice provides a new opportunity for membrane application since it does not require any purity increase across the membrane. Normally one would expect a purity rise across the membrane in the UF or NF applications. Unfortunately high separation efficiency is accompanied by low fluxes. Conventional purification processes can remove only 25-35% of total non-sugars, whereas the chromatographic separators remove about 85% of non-sugars and color bodies. Most of these non-sugars (e.g., monovalent ions) are considered “non-removable” by conventional methods. The separation that is difficult or impossible to achieve using membrane technology alone can be easily accomplished in combination with ion exclusion chromatography. The chromatographic process does not tolerate suspended solids in the feed stream. Therefore membrane filtration as a method for suspended solids removal and chromatography as a powerful separation tool seem to be the most efficient combination for purification of sugar juices and syrups.

Most of the existing industrial chromatography applications use the simulated moving bed (SMB) principle. An SMB system usually comprises one or several columns filled with separation medium, e.g., ion-exchange resins in separation of sugars. In the SMB process feed stock and eluent are continuously added to the system. The points of feed and eluent introduction and products withdrawal are switched periodically simulating countercurrent movement of separation media. By
varying the ratio between product fractions it is possible to build up an internal component inventory inside the system. Most importantly only fractions of the inventory are removed as products. The rest of components keep recirculating inside a closed loop. A principle flow diagram of an SMB process with eight cells is shown in Figure 1. Due to the multi-pass nature of SMB processes higher product purity and recovery can be achieved.

Continuous recirculation of components inside SMB system imposes strict requirements on suspended solids elimination out of feed streams. Backwashing of separator cells is considered as an emergency mode of operation rather than a routine procedure. Raw beet juice contains a very wide spectrum of suspended solids ranging in size from a fraction of a micron to several millimeters. Conventional processes can easily remove the coarser particles but only membrane technology may be a viable option for removal of submicron particles. Juice sterilization as a side effect of membrane filtration is expected to benefit downstream operation.

RESULTS AND DISCUSSION

In spite of continuous efforts of many researchers very little information has been published on the performance of microfiltration or ultrafiltration systems. A principle possibility of beet juice microfiltration was shown in the paper by specialists from Southern Minnesota Beet Sugar Cooperative and Dow Chemical Company\(^\text{14}\). Presented data are not sufficient though to make any conclusion about the feasibility of a new process. Paper presented by R. Kwok at the Sugar Processing Research Institute Workshop\(^\text{7}\) gives detailed description of a membrane application for ultrafiltration of clarified cane juice. After two years of operation the membrane filtration was still in the development phase.

Experimental results obtained on various sugar-containing streams at Amalgamated Research Inc. in Twin Falls, Idaho are discussed below. The purpose of preliminary testing was to evaluate a principle possibility of microfiltration and analyze feed and product streams. Therefore, tests were carried out with both concentrate and permeate recycled back to the feed tank. An average test lasted for about 3-5 hours. It was not advised to run longer tests in recycling mode due to possible changes in properties of a feed solution. Dow CMF 0.2 micron hollow fiber membrane with 1.5 mm bore diameter was used in all cases. Feed was initially screened through 500 micron sieves. Most of the tests were run at 70°C due to low temperature epoxy formulation on membrane tubesheet. Higher temperature was tested once to evaluate if higher fluxes can be obtained. Results are plotted in Figure 2. No flux optimization was done during the first set of tests.

Table 1 illustrates analytical data for beet molasses, raw juice and press water. Feed and permeate purity were analyzed polarimetrically, several samples analyzed for true purity by GC have confirmed the apparent purity data. Turbidity of the solutions was evaluated spectrophotometrically at a wavelength 720 nm. Dextran level was determined by E. J. Roberts method which accounts for both low and high molecular weight dextrans. Total hardness was analyzed by titration with EDTA solution. Level of the suspended solids was measured volumetrically by spinning a sample in the clinical centrifuge for five minutes.
An expected purity rise across a membrane did not exceed 0.5-0.6. Results look reasonable because most of the non-sugars are present in dissolved form and the size of their molecules is too small to be removed by MF or even "loose" UF membranes. Complete juice sterilization and suspended solids removal was achieved. Both color and dextran levels were reduced significantly in all tests. Apparently high molecular weight dextrans and color-forming molecules are rejected by MF membranes. This phenomenon may be explained by formation of a dynamic layer on the surface of a ceramic membrane. Late experiments with MF and UF membranes with pore sizes ranging from 200,000 MWCO to 0.2 micron showed insignificant difference in dextrans and color rejection. This confirms the theory that dynamic membrane is responsible for separation characteristics. Slight total hardness reduction can be explained by removal of precipitated calcium salts of organic acids. In general results appear to be very attractive from a technological standpoint. Microfiltration provides perfect pretreatment prior to ion-exchange softening and further chromatographic purification.

Cane molasses from various sources has been tested in MF experiments. Analytical results are presented in Table 2. Slight apparent purity rise was observed across the membrane. Suspended solids removal was adequate for chromatographic separator feed material. Usually cane molasses from various sources demonstrates significantly different properties, such as hardness, suspended solids, etc. Cane mixed juice treatment appear to be more technologically attractive solution. Suspended solids removal will be the only requirement for the membrane process. Certain screening should be required to eliminate bagasillo and large particulate matter which may plug the membrane channels. A chromatographic separator will do an excellent job of purification and color removal.

The second set of tests was carried out for two months during the 1995-96 beet slicing campaign. A fully automated membrane skid was operated in feed and bleed mode. Feed was pretreated using ARi proprietary technology. Pretreated beet raw juice contained about 0.3% vol. suspended solids and was screened through 250 micron sieve. Several MF membranes have been tested. Feed juice temperature was maintained at 90°C to minimize viscosity and prevent bacterial growth. Typical test results are shown in Figures 3 and 4. Dow CMF hollow fiber membranes with 3 mm I.D. showed good and stable performance without cleaning for 5 days at 120 GFD. High temperature epoxy tubesheet formulation was used in all tests. Several fibers were broken during the tests. Regardless of relatively good performance we do not consider hollow fiber membranes suitable for sugar juice applications due to the possibility of unexpected failure and replacement expenses.

With Ceramem membranes (2x2 mm channels) it was possible to sustain flux at 100-105 GFD for over two days with slow TMP increase over the test period. The plot in Figure 5 illustrates that TMP rise was not a consequence of membrane fouling. Some beet fibers were breaking through the screens and slowly accumulating between two modules in series. This phenomenon can be avoided with modified pretreatment.

Taking into consideration the size of potential investment and importance of the long-term testing we tested several membranes during the campaign of 1996-97. All tested membranes produce permeate of excellent quality sufficient for further chromatographic separation. Final selection should be accomplished by comparing capital and operating expenses for different systems. Since the cost of membrane replacement may be 30-60% of an initial capital investment, evaluation of a membrane
service life is extremely important. Therefore one cannot overestimate the importance of thorough long-term testing.

Results obtained above do not provide sufficient information to reliably design a membrane system. They rather illustrate the difficulties on the way to development of a “ready to apply” process and emphasize the need for a serious R&D program.

CONCLUSIONS

1. Possible applications for membrane technology in beet and cane sugar industry have been reviewed. The sugar industry appears to be a large potential market for membrane technology.

2. Use of membrane processes prior to chromatographic separators appears to be an ideal combination for both beet and cane juice purification. No purity rise, only suspended solids removal is required as a result of membrane filtration.

3. Analytical results are presented showing the effect of microfiltration on raw juice, beet and cane molasses, and press water. The data look very promising from a technological point of view.

4. Necessity of serious long-term testing is emphasized for projects involving serious capital investment.

5. Additional testing of industrial scale modules is required for final economic evaluation. An efficient cleaning program is yet to be developed, and concentrate and associated sugar losses should be evaluated.
REFERENCES


366
FIGURE 1
FLOW DIAGRAM OF AN EIGHT-CELL CHROMATOGRAPHIC SEPARATOR

Feed

Raffinate

Wate

Extract

1
2
3
4
5
6
7
8
Dow CMF, 3 mm, 0.2 mkm, periodic reverse flow
Conc.factor=3, SS=0.3-0.4 % vol., 90 deg C
Raw beet juice MF
Twin Falls, Jan 12-14, 1996

Flux, GFD
TMP*10, psi

Ceramem, 2x2, 0.2 mkm, periodic reverse flow
Conc. factor=5, SS= 0.3-0.4 % vol., 90 deg C
Two 1' modules arranged in series
FIGURE 5

Raw beet juice MF
Twin Falls, Jan 12-14, 1996

Time, hours

Feed inlet pressure, psi

TMP, psi

Ceramix, 2x2, 0.2 um, periodic reverse flow,
Conc. factor = 5, SS = 0.3-0.4 % vol, 90 deg C
### TABLE 1

Analytical Sheet  
DOW CMF TEST  
Feed material - beet molasses, raw juice, press water

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<th>Date</th>
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<th>AP (%)</th>
<th>pH</th>
<th>meq Ca/100 DS</th>
<th>720 nm abs.</th>
<th>Color</th>
<th>Solids, % vol.</th>
<th>Dextran *</th>
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* Dextran, ppm on DS (analyzed by E. J. Roberts method)  
** True purity by gas chromatography  
*** CFU/ml of feed solution at 75-80°C (cool feed numbers are much higher)
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* Dextran, ppm on DS (analyzed by E. J. Roberts method)