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# using Spiral and Tubular Polymeric Membranes.

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## Abstract

Koch Membrane Systems, Inc. (KMS) has been working with Amalgamated Research, Inc. (ARi) and their partners in the beet sugar industry in the US and in Europe since 1996. The primary focus of the study has been the integration of ultrafiltration membrane technology into ARi's raw juice purification system. The challenges for polymeric membrane filtration in this application include high temperature (185°F), an abrasive environment (approx. 2-3 % suspended solids), and constantly changing feed quality. KMS has been successful in the development of high temperature polymeric membranes in spiral and tubular configurations for sucrose applications and these membranes show the greatest promise of success in this application. Pilot systems have been operated in the United States and in Europe and the results of three seasons of testing with both spiral and tubular membrane configurations have been very promising. A commercial scale spiral membrane system with the capacity to treat approximately 5% of the factory juice stream has been in operation at ARi for two campaigns and is installed downstream of a clarifier and prefiltration unit. In addition, a tubular membrane system has been piloted to further concentrate solids and to increase sugar recovery. The membrane trials have focused mainly on optimizing system performance with respect to membrane productivity, cycle run time, cleanability, sucrose recovery and membrane life. The membrane productivity has been consistent at varying operating conditions even with repeated cleanings. Initial water fluxes and differential pressures have also been recoverable, indicating the absence of irreversible membrane fouling and plugging, respectively, in the spiral membrane modules.

#### Introduction

# NEMBRANE SYSTEMS

The typical Beet Sugar processing factory, as it exists today, consists of five major areas, as shown in the generalized block flow diagram in Figure 1. Raw juice purification focuses on the removal of nonsucrose impurities. These impurities occur in both true and colloidal solution with the colloidal substances making concentration of the diffusion juice as well as crystallization of the sugar contained in it very difficult. The use of lime and carbon dioxide in raw juice purification facilitates the removal of these colloidal impurities by coagulation and of other impurities by adsorption on the surface of the calcium carbonate crystals. Sulfitation, the addition of sulfur dioxide gas, after second carbonation serves to adjust pH and to reduce color formation in the thin juice.

This process has remained largely unchanged for the past several decades in the United States. In Europe, process changes have occurred in an attempt to lower energy costs, with consolidation of small factories to create larger, more efficient factories being a common tendency<sup>1</sup>. This consolidation requires plant expansion or more efficient utilization of the existing equipment in order to process the additional beet load.

In recent years, environmental issues have come into focus and work is being done to evaluate the possibility for a cleaner raw juice purification operation. The preliming, carbonation, sulfitation and lime kiln all incur large costs and are pollution concerns. The carbonation and preliming stages of the process produce large amounts of waste-lime slurries, which must be disposed of efficiently and inexpensively. Normally these slurries are either dewatered using filter presses and the filter cake sent to land-fill, or deposited in lime ponds where settling acts as the dewatering mechanism, and the solids are removed at the end of campaign. This provides the incentive to develop a method of purification that does not involve the addition of large amounts of chemicals, which require complex systems for preparation and addition, as well as systems for removal and disposal of the resultant sludges.

A system that uses membrane technology and chromatographic separation could serve to replace the conventional system and thus provide a reduction in costs and pollution<sup>2</sup>. Koch Membrane Systems, Inc. has been working with both cane and beet sugar companies for the past several years in an effort to develop a membrane system which is capable of operating in the harsh environments present in these factories without sacrificing performance or life of the membranes. A discussion of the place of membrane technology in these applications as well as some of the results obtained from pilot scale testing in progress is presented.



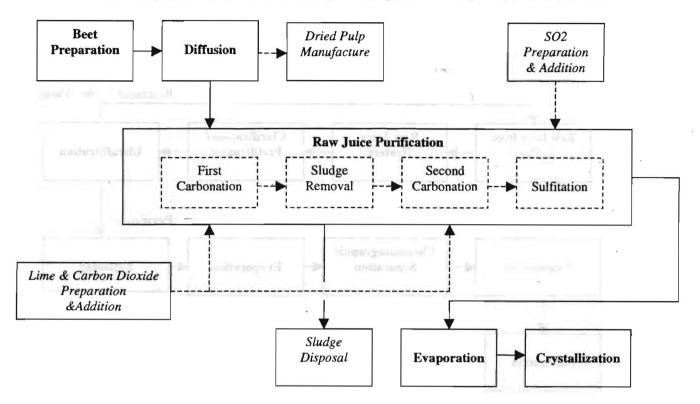


Figure 1: Beet Sugar Processing Factory

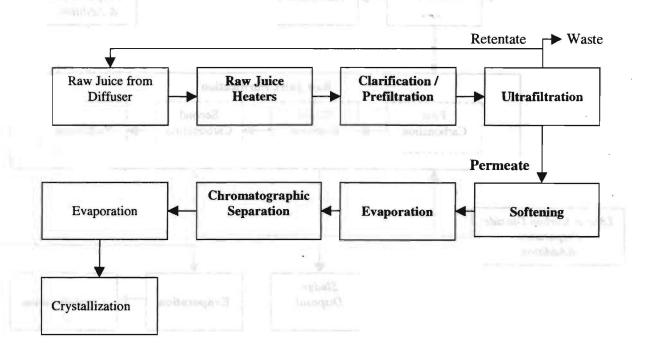
#### **Membrane Applications**

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Ultrafiltration as a Pretreatment Step for Chromatographic Separation of Raw Juice

There is a growing interest in developing a raw juice purification stage that would not rely so heavily on chemical addition and generate less waste, thus reducing the operating costs of this area of the factory. A possible solution to this problem would be the use of chromatographic separation of softened raw juice. Chromatographic separation is presently used in many beet sugar factories for the recovery of sugar from molasses. Amalgamated Research, Inc. (ARi) and others have been developing the application of this technology for raw juice purification, both in the cane and beet sugar industries.<sup>3, 4</sup> Research shows that a pretreatment system which incorporates fine filtration and juice softening by ion exchange is necessary to ensure the optimum performance of the separation technology. Ultrafiltration membranes have been shown to be capable of removing virtually all suspended solids from the feed stream and this ability

makes the application of such membranes here a valid one.<sup>4</sup> The overall process as proposed consists of pretreatment of the raw juice, using a clarifier and prefilter, followed by the ultrafiltration system, juice softening and evaporation before chromatographic separation. The juice is then ready to be returned to the traditional evaporation stage of the sugar factory, as shown in figure 2. (*below*)



#### Figure 2: Beet Sugar Factory incorporating New Technology

The potential benefits for this system include the elimination of the conventional raw juice purification process with its costly lime and carbon dioxide production and addition facilities and the associated lime disposal issues. It will also have positive environmental impacts such as reduction in factory odors, vent emissions and ammonia generation.<sup>5</sup>

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This new system could be installed while the factory continues its normal operation with only a short shutdown required for the final tie-ins to the existing systems. For these reasons, the chromatographic separation system could also be installed to increase factory capacity where the existing layout would make expansion of the existing raw juice purification system extremely difficult and expensive.

For the past two seasons, Koch Membrane Systems, Inc. and Amalgamated Research, Inc. have been running a commercial scale spiral wound polymeric ultrafiltration system with the capacity to treat approximately 5% of the factory juice stream at the Twin Falls, Idaho facility. The system treats the overflow from a pilot scale clarifier, which has been prefiltered using a 100-micron screen to remove larger suspended solids. This system has been operating at temperatures ranging from 80°C to 88°C. A prototype tubular ultrafiltration system has been installed to further concentrate solids and increase sucrose recovery.

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The membrane trials have focused mainly on optimizing system performance with respect to membrane productivity, cycle run time, cleanability, sucrose recovery and membrane life. The membrane productivity has been consistent at varying operating conditions even after repeated cleanings. Initial water fluxes and differential pressures have also been recoverable, indicating the absence of irreversible fouling of the membrane and plugging in the spiral membrane modules.

#### Ultrafiltration as a Means of Extending Campaign Length by Treating Raw Juice for Storage

Beet slicing campaigns last from 60 to 220 days of the year depending on the location of the factory. In Europe, the average campaign is 90 days; in North America, it is 180 days. Beet processors therefore have millions of dollars of processing equipment sitting idle for most of the year. Extending the utilization of the process plant has been a major objective in development of many new technologies in the recent past. Molasses desugarization, thick juice storage, etc., have all been means of accomplishing this goal. Add to this the trend towards factory consolidation which has led to smaller factories being shut down and larger factories absorbing their slicing capacity, and it becomes critical to consider methods of increasing factory capacity and optimizing use of existing systems in an economical manner.

One method by which this may be possible is to treat raw juice so that it may be stored for processing after the beet slicing campaign is completed. By facilitating this storage, the factory would be able to extend its sugar campaign without having to invest in large capital expansions.

Storage of raw juice has been attempted in previous years, in an attempt to minimize sugar losses during beet storage in warmer climates. This raw juice was limed and concentrated before storage. However excessive color formation and processing difficulties made this method unsuitable. It has been seen that concentration to above 67°Bx and pH adjustment to 8.0 pH will prohibit the growth and development of the yeasts and molds normally found in beet juices.<sup>6</sup> For juices of lower concentration, this was not the case. It has also been recommended that these juices be filtered before storage to remove heat resistant spores that could flourish during storage,

and also to remove particles that could act as nuclei to start crystallization. From the analytical data listed in Table 1 below, we can see that the ultrafiltration membranes remove all suspended solids as well as major amounts of polysaccharides and turbidity. The color content of the juice is also reduced. By its nature the ultrafiltration membrane is a positive barrier to bacteria, yeasts, mold and spores. Therefore it would appear that ultrafiltration of raw juice would make its storage more feasible, and thus facilitating the increased utilization of the plant equipment. A general block flow diagram of the suggested process is shown in figure 5 (*below*)

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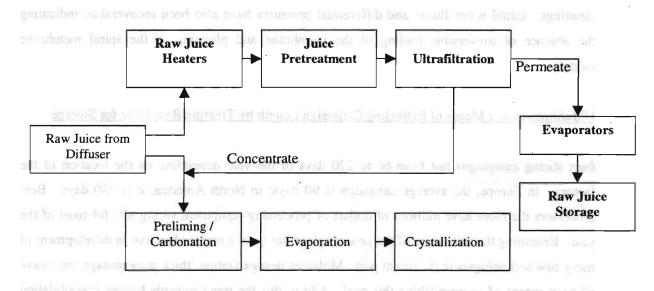


Figure 5 - Block Flow Diagram for use of Ultrafiltration in treatment of Raw Juice for Storage

In order to apply ultrafiltration membranes most effectively and economically in this application, one would not need to achieve the high concentration factors necessary for the chromatography pretreatment applications. A very economical system of spirals alone, operating at 2 - 5x VCF would be sufficient. Further, at these low concentration factors, performance of the membrane would be enhanced, with higher fluxes and / or run times to be expected because of the lower solids loading experienced in the system. Membrane life is expected to be longer in this application for the same reason.

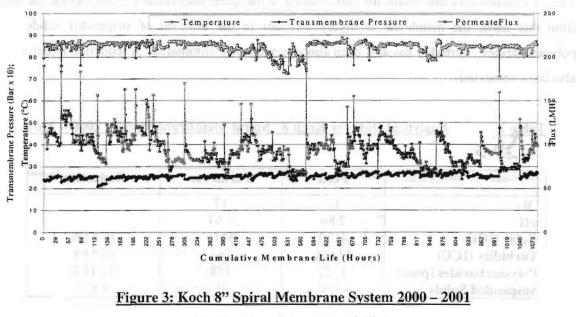
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#### Results of Membrane Demonstration

#### Spiral System

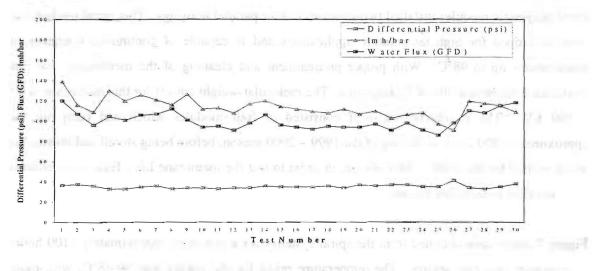
The spiral system in place is an 8" 4/2 commercial scale unit. It comprises eight 8" hard-wrapped spiral polymeric modules installed two-in-series in four parallel housings. This spiral module has been developed for high temperature applications and is capable of continuous operation at temperatures up to 98°C. With proper pretreatment and cleaning of the membranes, we can guarantee a membrane life of 2 campaigns. The molecular weight cut-off for this membrane is 45 – 100 KD. The membrane set itself consisted of four modules which had been run for approximately 300 hours at the end of the 1999 – 2000 season, before being stored and reinstalled in the system for the 2000 – 2001 season, in order to test the membrane life. Four new modules were installed to complete the set.

Figure 3 shows data obtained from the spiral system over a period of approximately 1100 hours of operation this past season. The temperature range for the testing was 80-88°C, with more consistent performance being observed at temperatures in the 85°C range. The average run time was 31 hours resulting in an average flux value of 100 lmh. The pressure drop gradient and Transmembrane Pressure (TMP) increase over each test were quite consistent with an average pressure drop rise of 6 psi and an average TMP rise of 2.5 psi, showing excellent repeatability of results.



Clarified Raw Juice, 14 - 18 °Brix

Figure 4 shows the water Flux data for the process runs included. After each cleaning cycle was completed, the system was run on water at a constant recirculation rate and the major process parameters recorded to measure the efficiency of the cleaning. The water flux was consistently returned after cleaning and, after the initial settling in period, the overall decline in water flux was negligible.



#### Figure 4: Koch 8" Spiral Membrane System 2000 - 2001

Clarified Raw Juice, 14 – 18 °Brix Water Flux Data

#### Analytical Data

Table 1 contains data that details the performance of the spiral membrane system. As can be seen from this table, the membrane is highly efficient in the removal of suspended solids and polysaccharides. Turbidity has also been almost completely eliminated. A reduction in color has also been witnessed.

Table 1:

Analytical Data for Koch 8" Spiral Membrane System, 2000 - 2001

	Feed	Permeate	Reduction
°Bx	17	17	2000 - 10 - 20 - 10 - 10 - 10 - 10 - 10
pН	7.86	7.64	
Color (ICU)	4150	3416	17.68%
Turbidity (ICU)	11124	161	98.55%
Polysaccharides (ppm)	3372	198	94.13%
Suspended Solids	<1 %	0	100%

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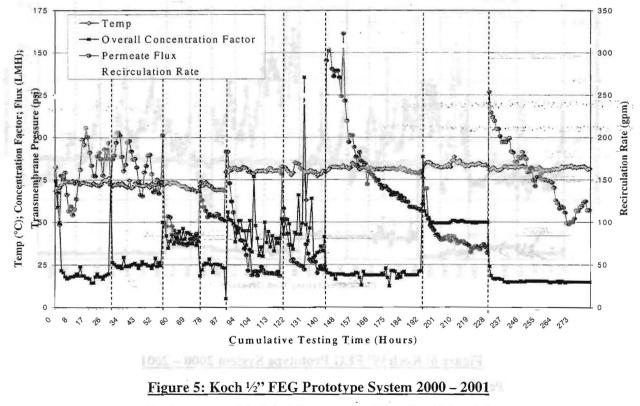
These data were acquired from samples taken late in the season, with the system being run at a volumetric concentration factor of 6X. Although we have not measured sucrose content here, it is expected, from previous studies, that the maximum purity increase across an ultrafiltration membrane in high purity juices will not exceed more than one purity point<sup>7</sup>.

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#### Tubular System

The tubular system in place is a prototype of a new configuration currently in development at Koch Membrane Systems. It consists if 98  $\frac{1}{2}$ " FEG tubes arranged in a two-pass configuration. The membrane on these tubes is the same as that in the spiral modules i.e. having a molecular weight cut-off of 45 – 100 KD. This prototype was installed new at the beginning of the season and is being tested both on unfiltered clarifier overflow and 5X retentate from the spiral system. The average temperature for all of these tests was 80°C, lower than the desired 85°C for this process.

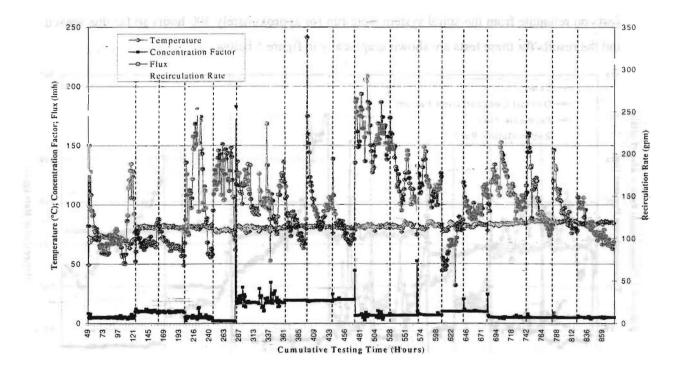
Tests on retentate from the spiral system were run for approximately 300 hours so far this season and the results for these tests are shown graphically in figure 5 below.

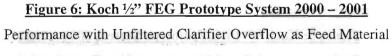


Performance with Spiral Retentate at 5X as Feed Material

The tests were split in half, with one half being run at 250 gpm recirculation rate, the other at a recirculation rate of 310 gpm. The process runs varied between 14 and 55 hours. As the chart shows, at higher recirculation rates for the system, productivity levels are also higher and these increases appear to correspond linearly with the increase in recirculation rate. Fluxes in the range of 25 - 100 lmh were recorded for volumetric concentration factors ranging between 20x and 50x.

The system has also been run for approximately 900 hours on unfiltered clarifier overflow and the results for these test runs are summarized graphically in figure 6 below. The tests, again, were split in half, with one half being run at 250 gpm recirculation rate, the other at a recirculation rate of 310 gpm. The process runs varied between 30 and 70 hours. Fluxes in the range of 50 - 150 lmh were recorded for volumetric concentration factors ranging between 2x and 20x were seen for the tests run at 250 gpm. For the runs at 310 gpm recirculation rate, flux ranges ranging between 60 - 175 lmh were recorded for volumetric concentration factors between 2x and 20x.





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#### Conclusion

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There are many potential applications for ultrafiltration membranes in the beet sugar industry and there have been many attempts to identify a membrane product that can solve the filtration needs of the industry economically. Working with ARi, Koch Membrane Systems, Inc. has been able to demonstrate the performance of polymeric membranes on pretreated beet diffusion juice over two campaigns. The membranes have provided consistent permeate quality throughout the test regardless of variations in the feed stream. The ultrafiltered juice is free of suspended solids, bacteria, yeasts and molds and shows significant reduction in turbidity and polysaccharides. These characteristics provide a juice quality that is necessary for feeding a chromatographic separation system, and also provide the characteristics necessary to meet the needs of juice storage.

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Installation of a membrane separation system in conjunction with other technologies provides an economic solution to the needs of factories requiring expansion. It is also believed that through the improvement of juice quality, membrane systems can be expected to provide a reduction in chemical consumption in the downstream processing of juice to make a final sugar.

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