MINERAL MEMBRANES FOR THE SUGAR INDUSTRY by Xavier Lancrenon (Applexion Inc., 15700 Lathrop Avenue, Harvey, IL 60426), M.A. Théoleyre (Applexion in Epone, France) and Guy Kientz (Rhône-Poulenc Inc.)

Ultrafiltration and Microfiltration are not new technologies. In some food industries, such as the dairy industry, these unit operations of separation have been used with success since the very early seventies.

The first generation of membranes was the organic. These membranes have been used successfully in many applications, where the characteristics of the products to be treated and the operating conditions are receptive to this type of membrane: low viscosity, low temperatures, frequent interruptions for cleaning, sanitizing procedures and membrane replacement.

For quite some time, those in the sugar industry have contemplated the possible use of membrane technology to replace conventional clarification and filtration methods--methods calling for heavy equipment and representing high operating costs, as well as creating those ever more difficult to resolve environmental problems.

Due to the characteristics of the sugar solutions to be treated, however, the first generation of organic membranes was not acceptable to the sugar industry. This industry--with its high temperature or viscosity of product to be treated, and a production schedule which does not tolerate interruption--demanded a new generation of membranes which could withstand these challenging conditions. Such membranes are now available--the mineral type, and these have been steadily gaining recognition since the beginning of the eighties.

This second generation of membranes is destined to become the ultimate performance/profit factor for the sugar industry of the future.

A COMPARISON OF ORGANIC VERSUS MINERAL MEMBRANES AND WHERE MINERAL MEMBRANES CAN BE SUCCESSFULLY APPLIED

The following table illustrates the differences between organic and mineral membranes in normal operating conditions. As can be seen, the mineral membranes represent a giant step in the potential use of membranes. This advance is due to (1) much higher operating pressures--up to 10 bars (145 psi); (2) no limit in temperature treatment for the conditions observed in the sugar industry (up to 350°C (662°F); (3) a life measured in years not months; (4) much greater resistance; and (5) the possibility of steam sterilizing for sanitization.

TABLE I COMPARING THE LIMITS OF ORGANIC VERSUS MINERAL MEMBRANES

	Organic Membrane	Mineral Membrane
Mechanical resistance --working pressure	2-5 bars (30-75 psi)	10 bars (145 psi)
Thermal sensitivity --working temperature	60 deg. C (140 deg. F)	No limit (up to 350°C or 662°F)
Lifetime --normal replacement	18 to 24 months	4 to 6 years
Normal cleaning conditions	NaOH 0.5% 60°C (140°F) HNO_3 0.5% 60°C (140°F)	NaOH 1% 85°C (185°F) HNO_3 1% 60°C (140°F)
Steam Sterilization	Not possible	Possible

There are several places in the sugar process where these membranes could partially replace the "conventional technology":

BEET SUGAR INDUSTRY

Carbonatation/Filtration: This method of purifying the sugar juice after diffusion has been almost the same since Napoleonic times. It requires very heavy equipment, such as the lime kiln, carbonatation reactors and the traditional filtration equipment. Moreover, it consumes approximately 4 kg % beet of lime rock and 0.3 kg % beet of coke.

The muds of carbonatation cannot be easily disposed of, since, unlike the organic nonsugar removed from the raw juice by the carbonatation--which can be considered a by-product having a certain value, this treatment adds calcium carbonate and diatomaceous filter aid, making it a cake with absolutely no commercial value.

By using *crossflow ultrafiltration* instead of the carbonatation/filtration system, the beet sugar factory would at this stage of epuration produce a perfectly clarified thin juice. This would probably obviate the use of the standard liquor filtration units common to the conventional sugar process. It would also produce a retentate of ultrafiltration which, after sweetening-off, could be incorporated directly in the pulp or sold separately on the market as a nitrogen-rich compound.

CANE SUGAR INDUSTRY

The most immediate application of *crossflow ultrafiltration* in this industry would be in the cane sugar refining: the clarification of melts of raw sugar or affinated raw sugar. Here again, *crossflow ultrafiltration* could replace a complete process block of the conventional technology--this depending on the refinery: either carbonatation followed by conventional filtration, or phosphatation followed by flotation and filtration. Both of these traditional methods create environmental problems which the *crossflow ultrafiltration* does not, since no chemical or filter aid needs to be added to the product.

For the production of liquid sugar from cane products such as cane juice, cane syrups or raw sugar, a full clarification is also required. For these special applications, *crossflow ultrafiltration* may also be the solution of the future.

In both the beet and cane sugar industries, *crossflow ultrafiltration* could be applied in specific cases to molasses or effluent treatment.

As can be seen from the above, the field of application is large and covers areas where the industry is accustomed to investing in heavy equipment.

INTRODUCING *CARBOSEP*™* AND *KERASEP*™* MEMBRANES

Table 2 indicates the membrane separation spectrum, showing the range for ultrafiltration (20 to 2000 angstroms) and microfiltration (0.1 to 10 microns, or 1,000 to 100,000 angstroms).

Carbosep and *Kerasep* mineral membranes are designed for ultrafiltration and microfiltration applications. For these two types of membranes, Tables 3 through 8 show the main features of the membranes, the range of membranes available, and the main features of the modules.

These two tubular membranes are constructed of materials totally resistant to chemical corrosion and offer a good compromise between surface area per volume installed and tube length, to obtain low energy consumption.

TABLE 2

Membrane Separation Spectrum

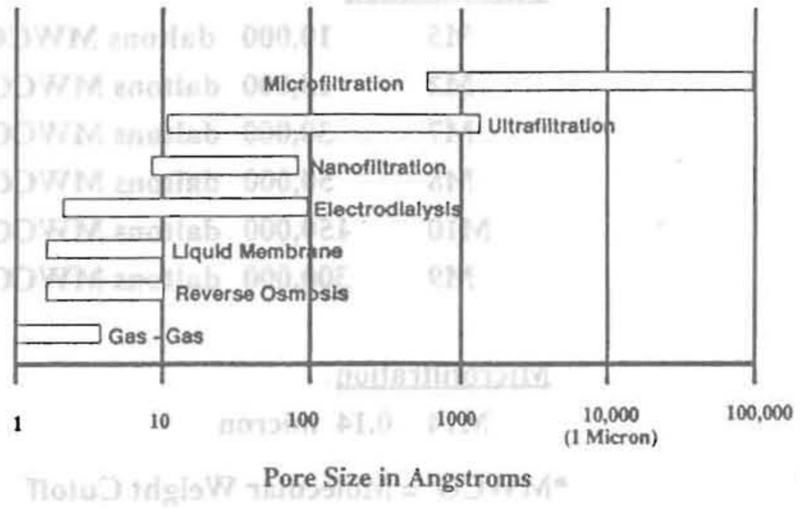


TABLE 3

Main Features of Carbosep® Membranes

Membrane Support	Sintered Carbon
Membrane Layer	Metallic Oxide
Layout:	
length	1200 mm/47.25 in
outside diameter	10 mm/0.40 in
inside diameter	6 mm/0.23 in
Mechanical Features:	
bursting pressure	40 bar/580 PSI
service pressure	15 bar/217 PSI
Chemical Features:	
service pH	0 to 14
steam sterilization	yes
chemical sterilization	yes
Process Temperature	
standard modules	up to 150°C/302°F
special modules	up to 350°C/662°F

TABLE 4

Range of Carbosep® Membranes

Ultrafiltration

M5	10,000	daltons MWCO*
M2	15,000	daltons MWCO
M7	30,000	daltons MWCO
M8	50,000	daltons MWCO
M10	150,000	daltons MWCO
M9	300,000	daltons MWCO

Microfiltration

M14 0.14 micron

*MWCO = Molecular Weight Cutoff

TABLE 5

Main Features of Carbosep® Modules

		Membrane Area		FITTINGS TC Clamp		FITTINGS Flange (DIN)	
Type	Number of Tubes	m^2	Sq. Ft.	Permeate	Retentate	Permeate	Retentate
S1	1	0.023	0.25	1"	1.5"	-	-
S7	7	0.16	1.72	1"	1.5"	20	40
S37	37	0.84	9.05	1.5"	4"	20	100
S55	55	1.25	1.35	1.5"	4"	20	100
S151	151	3.43	36.92	1.5"	6"	20	150
S252	252	5.73	61.68	1.5"	8"	32	200

TABLE 6

Main Features of Kerasep™ Membranes

Membrane Support	Monolith $A1_2 0_3/Ti0_2$	
Membrane Layer	$Ti\ O_2$ or $Zr\ O_2$	
Layout:		
length	1200 mm/47.25 in	
outside diameter	25 mm/ 1 in	
inside diameter	3 channels	10 mm/0.39 in
	7 channels	6 mm/0.24 in
	19 channels	3.5/0.14 in
	56 channels	2 mm/0.08 in
Mechanical Features:		
bursting pressure	50 bar/725 PSI	
service pressure	12 bar/174 PSI	
Chemical Features:		
service pH	0 to 14	
steam sterilization	yes	
chemical sterilization	yes	
Process Temperature		
standard modules	up to 150°C/302°F	
special modules	up to 400°C/752°F	

TABLE 7

Range of Kerasep™ Membranes

Ultrafiltration

15,000 daltons MWCO*
300,000 daltons MWCO

Microfiltration

0.1 micron
0.2 micron
0.45 micron
1 micron

*MWCO = Molecular Weight Cutoff

TABLE 8

Main Features of the Kerasep™ Modules

No. of Monoliths	Number of Channels per Monolith			
	3	7	19	56
1	0.11 m^2 1.2 ft^2	0.16 m^2 1.72 ft^2	0.25 m^2 2.7 ft^2	0.5 m^2 5 ft^2
9	0.99 m^2 10.8 ft^2	1.44 m^2 15.48 ft^2	2.25 m^2 24.3 ft^2	4.5 m^2 45 ft^2
19	2.09 m^2 22.8 ft^2	3.04 m^2 32.68 ft^2	4.75 m^2 51.3 ft^2	9.5 m^2 95 ft^2
37	4.0 m^2 44.5 ft^2	5.9 m^2 63.6 ft^2	9.25 m^2 100.0 ft^2	18.5 m^2 185 ft^2
99	10.9 m^2 116.8 ft^2	15.8 m^2 170.3 ft^2	24.75 m^2 267.3 ft^2	49.5 m^2 495.0 ft^2

Carbosep, with its carbon support, offers the widest range of ultrafiltration mineral membranes available to the industry today.

Kerasep is a monolith of aluminum oxide protected by a matrix of titanium oxide, which makes it absolutely nonsusceptible to high temperature alkaline attack (pH over 12, temperature over 70°C)--a condition which can be observed on conventional ceramic membranes. The very high porosity of this monolith allows a very high flux of filtration through it. It is, therefore, an ideal tool to employ for all microfiltration problems.

The system installed can be classified as one of two types:

CONTINUOUS, SINGLE-PASS

Figures 1 and 2 show a mono-stage and a 3-stage continuous, single-pass system. In this case, the retentate is extracted continuously and, therefore, the system works with a constant FCV (factor of concentration). The higher the FCV required, the higher the number of stages necessary.

BATCH

Figure 3 shows a batch system, where the retentate is sent back to the feed tank. This feed tank can, depending on the situation, be continuously fed with incoming product at the rate of permeate extraction--or simply contain the full load of product to be treated for a batch operation. In this case, the concentration in the retentate increases continuously throughout the run, until the required FCV is reached. This type batch system is often used for the final concentration of retentates and may be placed in series with a continuous, single-pass system.

EXAMPLE OF SYSTEM INSTALLED IN EUROPE BY APPLEXION FOR A NEW CORN REFINING PLANT

This system is designed to clarify corn syrups having the following characteristics:

DE	35 to 95
Dry substance	33%
Suspended materials	0.2% T.S.
Temperature	70°C (158°F)
Production time	24 hours per day

The loss in sugar through this clarification step is less than 0.5%.

In this particular case there are six identical lines of ultrafiltration: five in production with the sixth in cleaning or waiting, so production is continuous. There is no interruption of production for cleaning.

Each line is comprised of three skids, including, respectively, eight, six and six No. S252 modules containing tubes of M9 membranes. This means there are twenty modules per line, or a surface area of 114 square meters (1227 square feet) per line. FCV on each line is 30.

The retentate of the five lines in production is sweetened-off by diafiltration and concentration by means of another ultrafiltration line made of two skids (in series) containing, respectively, six and four S252 modules of *Carbosep* M9 ultrafiltration membranes. This amounts to an additional 57 square meters (614 square feet) of surface area for sweetening-off and concentration of sediment. The total surface area of this system is 741 square meters (7,977 square feet).

Cleaning is done in several sequences, using hot caustic soda and nitric acid. These cleaning procedures are performed once each day over a two hour period for each line of ultrafiltration.

FIGURE 1

SINGLE STAGE SIMPLE CONTINUOUS SYSTEM (ONCE-THRU)

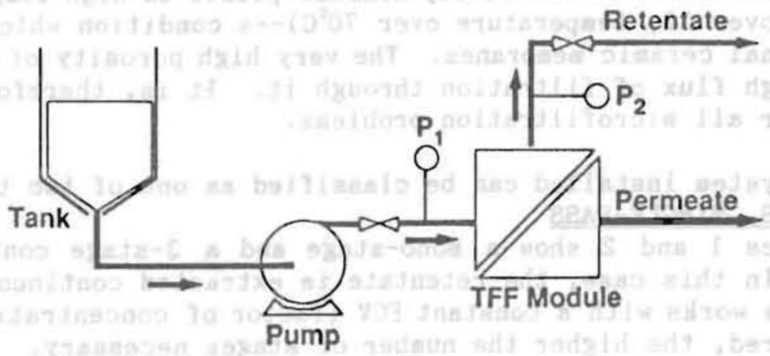


FIGURE 2

FEED AND BLEED BATCH SYSTEM WITH FEED PUMP

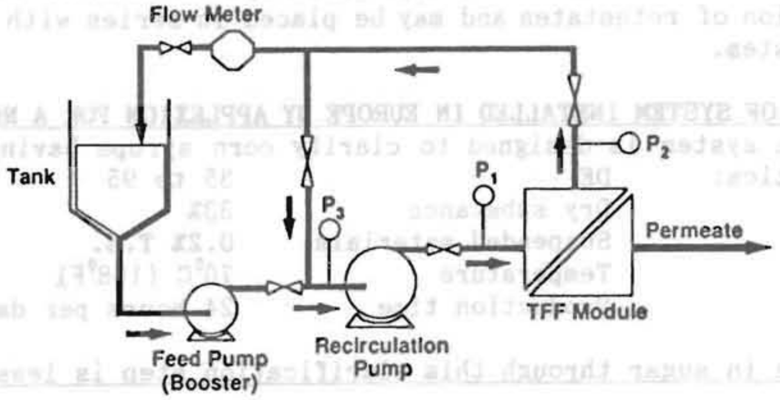
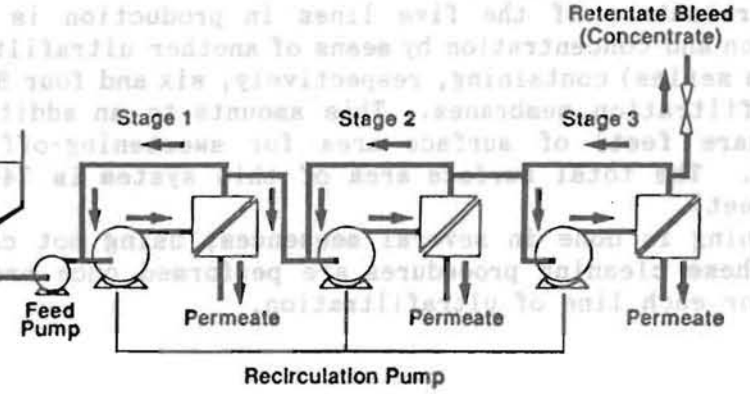


FIGURE 3

THREE STAGE FEED AND BLEED CONTINUOUS SYSTEM



The by-product, containing primarily fibers and proteins, is sold to be used in the manufacture of animal food.

This type of clarification will, without a doubt, replace conventional filtration systems using filter aid in modern corn refining plants.

LABORATORY TEST UNITS are available (to determine the most effective type of membrane for a particular application) as well as **PILOT UNITS** for use in gathering the necessary data for scaling-up a system. It is important to mention here that the *Carbosep* and *Kerasep* pilot units use membranes which have exactly the same dimensions, including, in particular, the same length as the final commercial unit. Therefore, the pilot will reproduce exactly the same hydraulic conditions as the commercial unit. Hydraulic conditions through the module for a given velocity and transmembrane pressure are the same on pilot and commercial scale, thus eliminating any risk of an unpleasant "surprise" in performance when the commercial unit is started.

For a new application, the pilot stage is absolutely essential, even if the lab tests have proven successful.

Commercial installations exist which demonstrate, for example, a multi-stage, continuous, single-pass system used for the production of WPC (whey protein concentrate) in the dairy industry; a unit for standardizing protein concentration in milk before manufacturing cheese (--this type of unit is very similar to what could be a system for standardizing starch slurry before manufacturing a starch derivative); and a unit for the continuous manufacture of acetic acid from alcohol. (This latter unit illustrates a batch system where the retentate containing the bacteria "mycoderma aceti" is continuously recycled to the fermentation tank.)

Applexion and Rhône-Poulenc believe that ultrafiltration and microfiltration by means of the *Carbosep* and *Kerasep* membranes represent a new generation of products which have the potential for revolutionizing juice and syrup clarification in the coming years.

Beet raw juice ultrafiltration tests have shown that permeation flux can reach steady values of 200 liters/hm^2. This brings ultrafiltration to the point where it should be considered as a very credible alternative to conventional carbonatation.

The sugar industry, together with its technology suppliers, should eagerly undertake the intensive developmental work which this science deserves.

---

* *Carbosep* is a registered mark of Tech-Sep/Rhône-Poulenc.

* *Kerasep* is a trademark of Tech-Sep/Rhône-Poulenc