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Pilot testing

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I Introduction

This paper will address the subject of pilot testing. How to do it. How to interpret the results. How to avoid the more obvious pitfalls.

Pilot testing usually calls for a lot of measuring and analyzing and the amount of data generated can be quite overwhelming. Nevertheless, that is essential if the data shall be of any value.

Pilot testing is done for several reasons.

1. To find out which process is the best suited.
2. To find out whether membranes or other processes can solve a problem in an economically way.
3. In case of a membrane process to find the best combination of membrane and system.
4. To find the best processing parameters.

II Before any testing

At least three points need to be clarified

1. Operation of a membrane filtration system is not free of charge. It will cost from US$ 0,20 and upwards per 1000 liter of permeate to operate a system. If that is considered too expensive, do not bother to test. The end user will not buy anything anyway.
2. Testing costs money. Often a lot of money. Membranes, equipment, shipping, travel and lodging, analyses and hours. It is essential to know Who-Pays-What up front. Serious companies have a budget. Not-so-serious companies do not want to pay for testwork. Beware of companies who want you to pay for all testwork. They are not likely to be serious buyers.
3. If a process shall be technical and economical possible the use of as well concentrate as permeate must be known. At least somebody must know that both streams are present and think about it. If one of the two streams is considered a major problem, e.g. due to BOD, then membranes will never be used.

There are too many examples on processes which are technical possible and which never materialize due the points 1 and 3.

- *Disposal of permeate from UF of cheese whey very nearly stopped that process in the mid seventies. The permeate contains 90 - 95 % of the BOD in the feed. Thus UF of whey it is a far cry from solving a pollution problem. But a use of lactose was found and the process is now well established and an essential part of any cheese manufacturers process lines.*
- *Recovery of water from textile dyeing effluent has so far been an almost complete failure. RO and NF works great. But legislation, technical problems with the disposal of the highly colored concentrate and fear of new technology means that only a couple of industrial spirals systems are in operation (February 2001)*

The argument for point 2 is often that “if the process works then the OEM will sell equipment to us and other companies and in this way get their money back.” There is some truth in that. But why should an OEM put money in development work for other companies?

The recent example is from the textile industry (February 2001). The end user droned away about all the plants we can sell in the future, when they successfully have commissioned their system and therefore we shall to a low prices. A minute earlier they told with pride that they had 40 % of the worlds market on polyester dyeing. That does NOT point to a lot of systems. Besides that the cost of fresh water for this particular factory is a factor of 10 higher (September 2001) than the price for recovered water. That actually makes RO quite attractive.

Few companies want in reality to share the results obtained. That makes sale of more systems to the same type of process unlikely.

The above means that a few minutes of thinking can prevent a lot of wasted time and money.

III What is the objective of pilot testing

Screen several membranes and find the best

Experience is a big helper when it comes to selecting membranes. It is rarely necessary to test more than two or three from one supplier. The end user may want to test membranes from several membrane suppliers and that increases the number of membranes significantly.

RO membranes are so standardized, that there in reality is very little difference between membranes from the four big players. Osmonics is special since it has membranes which can tolerate high temperature and rather extreme pH.

NF membranes are not born equal. Osmonics has true NF membranes, while most others supply loose RO. Desal DK and DL are world leaders in NF processing. In NF of water loose RO is more common, with the exception that DL is the only membrane doing a good job of sea water softening.

UF membranes differ a lot. Polysulfone (the whole family) is the workhorse in UF. PVDF is a good number 2. Osmonics has excellent and quite competitive UF membranes.

MF membranes are the most diversified. MF membranes may be made of several polymers, sintered stainless steel and ceramics. The Osmonics JX and JY (of PVDF) has some success.

Screen several membrane configurations and find the best

Spirals are used for many different applications. Also applications which were unthinkable a few years ago. But they are not the best for all membrane applications.

Spirals are superior

for RO and NF (solutions with no suspended solids).

Spirals have a large market share

in many process UF application. Hollow Fibers are competitive.

Spirals struggle

in microfiltration applications with solutions with suspended solids. (A few work fine, e.g. ED paint). Ceramic and hollow fiber membranes are strong.

Fiber systems have the last few years made enormous advances in the MF area. Especially MF of water, either as stand-alone or as pretreatment before RO. Very large systems have been built and larger systems are in building. Fibers can be backflushed and that is one of their main advantages.

Plate and frame systems have a few exclusive uses. This type of system is mostly sold too expensive. Mid 80-ties plate and frame dominated the food and dairy market. They were wiped out by spirals. Newer types struggle to regain some market share. The membrane area in operation is now <1% of the membrane area.

Tubular systems has a very limited market. It is really a first generation type of system. Tubes one advantage is tolerance to all types of suspended solids, including fibers. Price, energy consumption, space demand plus a bit more has almost made it extinct. PCI will probably disagree and they have made a good job of finding applications and markets willing to pay their price. However, the membrane area installed and in operation is sliding into the <1% range of membrane area of the world.

Ceramic membranes have a few uses. They have heavy competition on price and performance from fibers. Note that the price for ceramic membranes (September 2001) is dropping fast, thus making them more competitive.

Screen various pre-treatment possibilities

Pretreatment is a very big subject. However, the objective is simple: prevent precipitation of solutes and prevent excessive amounts of suspended solids from entering the system.

See section IV in this paper and see also Membrane Filtration Handbook for a more detailed description.

Heating. See section V.

A brief summary of pretreatment is s follows.

Filtration. The feed to a membrane system is always filtered. The filter micron range depends on the membrane system and product to be treated. The most course filters are 50 to 100 micron. It is very common to use 5 to 10 micron filters. It is rare to have tighter filters, although UF may be used (0,02 micron).

Whey and milk is not filtered at all. It is treated in a centrifuge. A 100 micron security screen is all there is.

Pharmaceutical products are often treated on 5 micron absolute filters.

Oil emulsion are often filtered on a moving band filter.

PH adjustment pH adjustment is commonly done to prevent precipitation, primarily of Ca-compounds. Use of strong mineral acid such as sulfuric acid is quite common. A more exotic product (and quite difficult to handle) is CO_2.

The pH of sweet whey is often changed from 6.2 to 5.8 to minimize Ca-phosphate precipitation.

Evaporator condensate often get pH increased to around 7.

A textile degreasing effluent get pH decreased from 11 to 9.

Heating/cooling Industrially it is quite common to heat or cool. The objective can be to increase solubility by heating , e.g. Carrageenan, or increase solubility by cooling, e.g. CaCO3. It can also be desirable to achieve an operating temperature which is bacteriological safe. Only in case of water treatment is heating done in order to maximize capacity.

A boiler feed water RO plant is typically running on water which is heated to around 30 deg C

Many food products are cooled to 15 deg C or less.

Carrageenan is treated at >85 deg C - after being cooled from the boiling point.

Operating conditions.

Cross flow, operating pressure and operating temperature are the most common variable parameters. However, they are not quite freely variable.

Flow must be kept with limits. If flow is too low fouling may be excessive. If too high the pressure drop may be detrimental to the elements. *Example. An 8" element with 50P spacer needs a minimum flow of 6000 lph and can sustain a maximum flow of approximately 22000 lph. Industrially the flow is kept close to the maximum. By water desalination flow is often close to the minimum.*

Pressure It is a good rule to stay well below the maximum pressure stated by producers. The osmotic pressure may dictate that pressure must be well above that value. In reality the operating pressure is always somewhere in between those two values.

Industrial operating pressures (not valid for water treatment)

RO : Typically between 15 and 35 bar. Rarely in the range 40 to 70 bar and even more rarely between 7 and 15 bar.

NF : typically between 10 and 30 bar.

UF : typically between 2 and 7 bar, Very rarely above 10 bar.

MF : always below 5 bar. Best below 2 bar (actually 1 bar)

Determine flux as function of pressure with all other parameters kept constant. It is mostly fine to take readings after 30 minutes of constant conditions. If there is a maximum flux stay well under that value when designing an industrial system. The odds are that a large system will operate at lower flux than you can do in a lab.

Flux is particular tricky since there can be long term effects that you do not see for weeks or months. For some (all???) products there is something called “The Critical Flux”. When a system operates at this flux or lower it will be able to operate for very extended periods of time without cleaning or significant drop of flux. A now classical example is UF of surface water with humic acid, where 15 lmh has been used for years as a safe flux for CA membranes. That value is now creeping up to 22 lmh with newer types of membranes.

Do not expect a high flux in a spiral system. There are ceramic systems and special flat sheet systems (CR-filter) where several hundred lmh is common. For most spirals expect 15 lmh and be happy if it is 25 lmh in a process application. By brackish water RO 30 - 35 lmh is not uncommon. If testing shows >50 lmh do not believe it unless you are very sure of the conditions.

If temperature can be varied keep everything constant. Start at low temperature and increase temperature in small steps , e.g. 5 deg C intervals. Heat to the highest temperature which the product will tolerate.

The vast majority of membrane systems operate below 30 deg C. Some work close to 50 deg C. Very few (including spirals) operate in the range 50 - 90 deg C.

Once the best operating parameters have been established more elaborates test can be done to get real data for flux, rejection, fouling and cleaning.

By all testing keep very clear in our mind what the product can tolerate and what an industrial membrane system can tolerate.

Cleaning

Testing will at best give a hint about the cleaning problems of a real plant. All too often a plain water flush restores the membranes after a short test run. In an industrial system a much more elaborate cleaning is needed.

Discuss with Johnson-Diversey and Henkel. They can mostly give good recommendations for a cleaning regime.

The standard full blown cleaning regime can be

- Flush with water
- Alkaline cleaning — e.g. 1% Divos 108 or Ultrasil 110, 45 - 50 deg C, 30 min.
- Flush with water
- Acid cleaning — e.g. 0,5% Divos 2 or Ultrasil 76, 25 - 30 deg C, 20 min.
- Flush with water
- Alkaline cleaning — e.g. 1% Divos 108 or Ultrasil 110, 45 - 50 deg C, 30 min.
- Flush with water
- Disinfection — e.g. 0,5% Na-bisulphite at pH <4,5 or heat to 90 deg C.
- Flush with water

A full cleaning may last 2 - 3 hours. It is often possible the have a shorter cleaning. If for example you can start with acid, then cleaning step 1 and 2 can be skipped.

A rarely used method to cut down water consumption and cleaning time is to build a system which can be drained fast and efficient. That can e.g. be achieved by placing the housings vertically or at least slanted. It is a pity that such a simple and efficient approach is used so little.

IV Pretreatment

Pretreatment is such a large subject that I shall only make a few general remarks. The simple basic rules to follow are:

- Remove suspended solids, particularly fibers
- prevent solutes from precipitating

About suspended solids. Suspended solids comes in several shapes and forms.

Fibers	Found in quantity in Pulp & paper industry, Textile industry, Laundry waste water. Can only be handled in quantity in open flow channels, primarily tubular. Can not be handled by spiral wound elements
Platelets	Very small flat items. Seen as silt (clay) in rivers. Especially in the spring in the colder climates where flow in rivers increase dramatically, moving stones chipping each other. Is by all reported as exceedingly difficult to handle since platelets “glue” themselves to a membrane surface.
“Sand”	Small, round and hard items which can literally roll over a membrane surface. Finely ground sand. Pigments. Can be handled well even in spirals. E.g. ED-paint.
Hydroxides	“Wet”, slushy products which ranges from the colloidal to large aggregates. Can blind any membrane in any system. High shear, frequent backflush and frequent cleaning may keep a system running.

It is far too easy to pretreat during test conditions since the volume is limited. It is a challenge to test a realistic pretreatment scenario since the equipment necessary for industrial use may not be available in small scale. Discuss closely with the end user what kind of filters he uses and discuss with filter companies what they can recommend. The biggest danger is that the pretreatment during testing may be a far cry from reality, meaning far too good.

For large applications such as water desalination and whey processing the pretreatment is quite standardized. For most other processes pretreatment must be discussed with the end user. Precipitation, flocculation, sedimentation, heat treatment, centrifugation are a few of the processes used somewhere. See Membrane Filtration Handbook for more details.

The type of pretreatment and the time between cleanings is closely related. RO of water and UF of whole milk are two extremes.

- It is common to operate a water plant for months without cleaning.
- It is common to clean a milk plant every 8 to 10 hours.

This means that the pretreatment of water is very elaborate and may involve a lot of chemicals, while the milk pretreatment is approaching a rudimentary filtration.

Filters of many kinds are almost always used. Besides that centrifuges are common in the food related industries.

The micron range of pre-filters is a much debated item. Every industry and membrane producer has its own rules. By RO of water 1 to 5 micron filters are recommended and even MF or UF may be recommended. All the specification sheets I have seen does in reality reflect the conditions for RO of water

However, filter pore size and interval between cleanings is related. Industrially much more open filters are used. One simple reason is that you may alter the composition of the feed if the filter is too tight.

A few recommendations for industrial processes can be stated. Use them with care.

1. *Prevent at all costs fibers of any kind to enter a spiral.*
2. *Allow suspended solids if it spherical. e.g. pigment in ED paint.*

3. *A very crude rule of thumb says that the filter pore size shall be less than 10% of the spacer height in a spiral wound element. This is a quite controversial rule, which is far from being generally accepted.*
    a. *50 micron is mostly adequate for elements with 30 mil (0,7 mm) spacer*
    b. *100 micron is mostly adequate for elements with 50 mil (1,2 mm) spacer*
    c. *200 micron is mostly adequate for elements with higher spacers*

V Variables

There is in reality not so very many parameters to work with. Pressure and temperature are typical variables. The flow can only be varies within narrow limits. Furthermore, it is necessary to briefly discuss the difference between RO/NF and UF/MF. (See Pressure section)

Temperature.

Heating and cooling before membrane filtration is done but not too often. Few companies want to get involved in shifting large amounts of energy. It is expensive and best avoided.

The general rule is that higher temperature gives higher flux. One consequence is to choose the highest possible temperature in order to maximize flux and minimize pressure.

Flux increases by 2,7 to 3,3% per deg C, depending on the type of membrane and absolute temperature. However, that is only strictly true for processes where there is no precipitation on the membranes, e.g. water desalination. For most processes 1% per deg C is more common and precipitation can cause the flux increase to be zero. In a few cases flux drops when temperature increases.

Not all products can be heated and heating is generally considered too expensive. That means that the operating temperature is given by the process and it is not a variable. Keep than in mind when you test.

Furthermore, when testing KEEP CONSTANT TEMPERATURE. Keeping temperature constant may sound simple but it is not. A multitube heat exchanger can do the job. It just has to be an integral part of the system. Heating due to energy input from the pump(s) can cause the product temperature to increase to unacceptable levels. Since it is desirable to keep constant temperature heating must be avoided. In a few rare cases it is actually necessary to heat the product during processing.

Pressure.

See Membrane Filtration Handbook. Here is a brief summary.

NF and RO	Flux increases linearly with the NDP = Operating pressure - osmotic pressure. Typical range of operating pressure 10 - 60 bar.
UF	Flux is largely independent of pressure. Typical range of operating pressure 1 - 6 bar.
MF	Flux MUST be kept low. Otherwise severe fouling is certain. Flux and pressure may literally be inversely proportional. Typically 0,4 to 1,5 bar TMP is best. Constant TMP is by far the best. Very few membrane systems can do that.

Comments about Pressure and Flux

NF and RO	You may operate with constant flux, by varying the pressure. However, in the process industry it is not without perils
UF	You can rarely operate with constant flux, by varying the pressure. Water treatment is one of the few applications where it is done. And even here it is only technically healthy when nothing goes wrong. Operate at constant pressure and monitor flux.
MF	Do not even try to keep constant flux. That is doomed. Flux is what flux is.

VI Where to test? Own laboratory or by costumer?

You may work in you own lab for very small scale screening.

Go to the end user for serious testing. There you find lots of fresh feed and (usually) good laboratory facilities. They know at least what to look for and how to evaluate the permeate and concentrate.

Testing on site also helps the end user to understand the process and to gain confidence and knowhow. This is essential for the end user. Few people want to buy what they believe is brand new technology. Once an end user is happy with the equipment and the process then he is much more likely to buy.

Another side effect may very well be that the end user start to use the pilot equipment for other products and this almost invariable leads to new applications, found by the end user. That is an ideal for Osmonics. It is (2003) increasingly common that end users develop processes which Osmonics does not even know about.

Dairy products, fish products and other food products changes within hours. This makes it impossible to seriously test anywhere but by the end user.

Precipitation in pulping effluent like spent sulfite liquor and Kraft Black liquor will irreversible change to composition.

Oil emulsions may be unstable and form a layer of free oil.

VII Operation mode. Batch, Fed batch, Continuous

An industrial system is almost always operating continuously. A lab-system is almost always operating in batch. Only quite large semi-commercial test systems can operate continuously. This is not really an issue as long as all test data are noticed carefully and manipulated mathematically properly.

In many respect it is best to operate in batch. However, see next section about errors. Most flux and rejection data can me monitored very well. The one thing that batch operation does not show clearly is flux development as function of time. That can only be monitored by continuous operation.

Fed batch is a simple and handy way to operate, especially when the volumetric concentration ratio is high. Its main drawback is, that it is difficult to interpret the data and that the math involved is not commonly known. However, it is there. See Membrane Formula Handbook.

Continuous operation give by far the best data. All questions concerning flux, flux development and rejection coefficients can be determined.

Water desalination and all larger industrial systems operate continuously. The exception is the pharmaceutical industry which may go for batch operation in order to have a clear track of every batch.

Systems treating <1000 lph are mostly batch systems.

Systems treating >10 m3/h are mostly continuous systems.

It is recommended to look in the Membrane Formula Handbook. That may save a lot of calculation time.

VIII Known errors during test work

Pretreatment. *See section 4*
Duration of test work. *See next section*

Treated volume per unit of membrane area.

Let us assume that

- flux 25 lmh
- 22 hours of operation
- volumetric concentration ratio of 10.
- 3838C-30D element with 7,4 m²

This means that the amount of liquid treated per element in 22 hours is

$$25 * 22 * 7,4 * 10 /9 = 4500 \text{ liter } (= 1200 \text{ US gallon})$$

So in order to make a correct test 4500 liters (1200 US gallon) of fresh feed must be available. That is a lot, especially if the product is expensive. Nevertheless that is what it takes to make a realistic production run.

Treated volume per unit of membrane area and fouling

The fouling observed will not be realistic unless the correct volume per m² membrane area. The calculation above give a guide line how a realistic volume can be calculated.

An almost classical error is to establish total recycle of permeate and concentrate and keep recirculating for hours or days. This is absolutely unrealistic and will not show anything about flux and flux development. The reason: In raw feed there is a certain amount of product which can foul membranes. Once it has settled on a membrane no more precipitation takes place, even if the product is recycled for days or weeks. Only the correct volume of raw feed will foul the membranes with a realistic amount of “dirt”.

Feed quality and feed composition.

1. Testing at a facility usually means that the supply of fresh feed is not a problem.
2. If product is shipped it is almost guaranteed that it alters or ages. Growth is normal and almost unavoidable. The change of the product can be anywhere from negligible to very drastic. Only experience and discussion with the end user can give a hint to what extent a product changes with time. Since any change will influence rejection data and fouling characteristics this issue must be taken very seriously. Unfortunately it is rarely possible to quantify what the change or ageing does.
3. A batch of 22 hours of operation may subject the product to time/temperature conditions or to shear conditions it can not tolerate. In that case continuos operation is mandatory, even if it is considered difficult. Most food products fall in ths category.
4. All data generated relate to the past and there is no guarantee that the feed of tomorrow is like the feed of today. Only the end user *may* have the knowledge about this issue. This makes it essential to test as long as possible in order to experience most of the variations which can occur. However, it is a fact that surprises can pop up, no matter how long testwork is made.

Element size

Testing is often done with 2½”, 3,8" or 4" elements. Be very much aware that elements with smaller OD is more robust than elements with a larger OD. Choose the operating conditions in such a way that they reflect what can realistically be done in an industrial system.

It is recommended to use elements with the same OD as an industrial system, if at all possible. That will eliminate a lot of discussions later.

Pressure drop is a key parameter. Do not exceed 0,7 bar during testing if 8" elements are to be used industrially.

The most common rules are:

Water type elements	2½", 4,0", 8,0"	Maximum 0,7 bar per element
Dairy type elements	3,8", 3,9", 5,8", 6,3"	Maximum 1,0 bar per element

Cross Flow

It is often so easy in lab equipment to vary flow. Make sure to select a flow which is realistic for an industrial system

IX Duration of test work

Testing shall preferably be quite long, meaning weeks or months, in order to generate reliable data. Economic restrictions may limit that. However, weeks of testing is necessary if larger systems shall be built based on the test results.

A screening test is an exception. When the objective is to find rejection data only it is acceptable to take readings when the permeate composition is stable.

It is a common error to operate only a few hours. Test work of only minutes have been reported. The latter is simply pure nonsense since it typically takes 30 - 60 minutes before a system is stable.

A realistic test run shall preferably be of the same length as a real production run is. This can typically vary from 8 to 22 hours and may be as long as one week. That is the only way to find out how flux develops over time and to avoid at least some surprises.

- Growth of bacteria can give a few surprises after 6 to 8 hours.
- Precipitation can harden after many hours, to the point where cleaning is impossible.

X Mathematical data manipulation.

Generate flux curve(s) of a variable

By RO of water and a few very pure solutions flux can be calculated from physical constants, e.g. osmotic pressure, operating pressure and temperature or ionic strength. These fundamental calculations will not be discussed here, since that is documented in textbooks from universities. Be aware, that commercial programs such as Winflows and ROSA use a mixture of theoretical facts and experience to calculate flux and composition of the permeate and concentrate.

In order to use test data for calculation of flux it is necessary to make regression analyses. You can of course draw a flux curve on a piece of paper and get a good idea. However, that does not help you to make good calculations.

Permeate flux can be calculated as function of quite a few variables. It is common to use

I. Concentration of a particular solute, e.g. high MW component (protein) or low MW component (lactose)

II. Volumetric concentration ratio (or volume fraction)

The use of solids makes the data more generally applicable and they can with a bit of luck cover situations not covered by the test work. It also makes the formulae less well known. However they are there.

The use of Volumetric Concentration Ratio (sometimes called VCR) or the reciprocal of VCR which is Volume Fraction Concentrate (sometimes called X) is straight forward. However, the generated formulae are only applicable when the feed composition is quite constant.

VCR	= (Volume of feed) / (Volume of concentrate)	VCR >1.
X	= (Volume of concentrate) / (Volume of feed)	0<X<1.

You can generate several types of flux formulae.

- **Linear** Y = A + B * X.
- **Logarithmic** Y = A + B * ln(X).
  Any spread sheet can do that nowadays.
- **Hyperbolic** Y = (A + B * X) / (1 + C *X).
  The hyperbolic function is excellent since it describes physical phenomena very exact, e.g. viscosity of water. Since it is a non-linear function it is hard to find the formulae for the curve fitting. Furthermore, NEVER extrapolate unless you have checked how the curve behaves. The curve goes through infinity for X = -1/C and unless you are aware of that you can get the strangest results. The worst is that you can get a result where an error message would be appropriate.
- **Polynomium** Y = A + B * X + C * X^2 + D * X ^3 .....
  It is recommended to stay away from a polynomium, especially higher degree. They can fit the measured data wonderfully but also in a way which bears no resemblance to the real physical world.

Note that there exist formulae for calculation of the average flux as function of X and solids. Very few know these formulae and they are a bit complicated. See Formula Handbook.

The writer has programs for WINDOWS (and for DOS) for all these calculations. Ask for them. They can run under Windows 95 through Windows XP.

Average flux and instantaneous flux

Average flux and instantaneous flux is quite similar to average speed and instantaneous speed. Driving from one town to another you first keep an eye on the instantaneous speed in order not to get a ticket for speeding. After you arrive you can calculate the average speed from the distance traveled and time used.

Similarly, it is easy to measure the flux at any given time. All you need is to measure a volume per time unit, e.g. ml/min. That can easily be transformed into lmh. This flux is called in instantaneous flux. When you start the flux is high because the membranes are clean and the TDS is low, meaning high NDP. When you finish membranes are dirty and TDS is higher than at the beginning, meaning low NDP. When you have reached the desired TDS or VCR, it is fairly easy to find out what the flux is in average as LMH, at least if you operate as true batch. If you operate in fed batch or continuous it is a lot harder to calculated the average flux.

The instantaneous flux is fine. However, it is rather useless when it comes to finding the number of m^2 of membrane needed in an industrial system. So unless you have batch data you need other formulae to be able to calculate the average flux.

Generate rejection data

Rejection is used a lot. What you in reality measure is % permeability as 100 * (concentration in permeate) / (concentration on concentrate). From that the rejection is calculated as 100 - % Permeability.

Rejection (or permeability) of solutes can be a function of many variables, such as concentration, ionic strength and temperature. The first guess is always that permeability is constant. Test data will show whether or not that is correct. You can at any rate generate the same types of formula for rejection as for flux.

Generate composition data

Composition of permeate and concentrate is essential. Examples.

- By RO of water the content of salt in the permeate is a key competitive parameter
- By UF of milk and whey the composition of the concentrate is essential for the process.
- By RO of condensate the BOD of the permeate is an important parameter.

Often such data and know-how is confidential and only known inside a few companies.

NOTE 1.
Work in W/W and not in W/V or V/V. Only by W/W will the calculations be reasonably correct. When TDS is <10% is makes little difference. However, ever increasing demands to warranty and yield of product can make a small error in TS recovery very expensive indeed.

NOTE 2.
Be aware that two phase systems. e.g. oil-in water, need special attention when you make a mass balance. In order to work exact you need to do calculations on the water phase *and* the oil phase.

A very actual example is UF of whole milk. The whole milk contains around 4,5% fat. A 5X concentration will produce a concentrate with more than 20% fat. It is essential to know where the solutes are to avoid grave miscalculations of the composition of the concentrate.

- Divide the feed in a water phase and a fat phase
- Do all calculations for the water soluble solutes in the water phase.
- Combine oil and water phase to get the final concentrate.

If you forget that there is 20% fat in the concentrate you have 20% less (as kg) water soluble solutes than you believe. This is further compounded by the difference between V/V, W/V and W/W. Assume that you are a cheese producer. It makes a very serious difference when you expect to get 20% increase in kg cheese production and you in reality only get a very small fraction of that.

XI Design of full scale systems based on test data.

Scale-up factor

It is common practice in chemical engineering never to scale up more than a factor of 10. Nevertheless, it is also a rule rarely followed in membrane technology. The shear volume of product needed to do good testing and the costs involved can be prohibitive. On the other hand, it is lunacy to build a large system for an expensive product if you can not afford to pilot it correctly.

After some 30 years of industrial use of membrane there is some know-how in several applications, which may legitimate small scale testing or even no testing.

- Desalination of water by RO is so well documented that on the basis of a good water analysis testing is not needed.
- Nanofiltration of water can sometimes be predicted with some accuracy.
- RO, NF and UF of dairy products such as milk and cheese whey can be predicted quite well by qualified OEM's. This knowledge is proprietary and not common knowledge.
- UF of some types of water, e.g. removal of humic acid, can be predicted well.

The four membrane processes can be predicted theoretically as follows.

RO Models are available. They can with some accuracy predict the behavior of products not yet tested.

NF Poor models are available. It is in reality impossible to predict the behavior of new products.

UF Models are available. However, they can predict just about nothing and only experience may be of help.

MF No prediction possible - yet.

When asked about a new product the standard answer is still "We have to test it".

Safety factors

There is always a battle between the technical safe and the economical possible. If good testwork has been done I recommend to add at least 30% membrane area to compensate for errors. One reason is, that 8" elements rarely achieve the same flux as 2½" elements, even when they seem to operate under the same conditions. You can almost say that the efficiency of 8" elements is only 75 - 80% of 2½" and 4" elements.

It is highly recommended to build systems in such a way, that it is easy to add more membrane area and to handle volumes different from the predicted. Use one or more of the following suggestions.

- Make room for and include connections for more housings.
- Do not fill the housings completely. E.g. only 5 elements in 6-element housings. Alternatively install some empty housings. (Look out for flow and pressure drop.)
- Make sure that pumps, valves and controllers can handle more volume and/or pressure.
- Make sure that CIP equipment is adequate.

XII Final remarks - Why are test data never exact?

Testing is necessary to generate data from which a plant can be designed and costing can be made. No matter how long time you test and how accurate you measure there will be some inaccuracies and changes. Membranes change or age, products change, operators change, demands change etc. There are so many variables, that the writer expects any set of data to quite inaccurate. So in the end, all you can do is to make an educated guess. One can hope that it one day will be possible from theoretical knowledge to predict how membranes work. However, a major breakthrough in membrane science is needed to get that far.

Use your common sense when you design a system, and never forget the phrase coined by Mr. George Hutson, Filtration Engineering:

"There is no substitute for a big membrane area"

Test/product form (testing at own facility) Date : #

Initials : # Responsible # Test number : #
Agreed price for test : #

Product shipped : # Received : #

Requested date for testing : #
Customer present? : # Book hotel room(s): #

Company : #
Street and number : #
Town, ZIP, Country : #
Contact person(s) : #
Telephone : + Fax : +
e-mail : : # WWW :

Product description : #

Use of product : #

Safety : #

Storage information : #

Description of test and product
Process : RO # NF # UF # MF #
Element type : # Membrane type : Element area : #
Maximum allowed temperature, deg C:
Conductivity, microS : pH : deg Brix :
Volume received : # Volume used : #
Process type:
Clarification Concentration Separation Desalting Other
Requested specs for permeate and/or concentrate (TDS, % HMWC, Brix, microS)
Concentrate : #
Permeate : #
Pretreatment before membrane filtration:
Estimated operating parameters
Feed Pressure ,bar : # Feed Flow, lph : # Temperature, deg C : #
Diafiltration volume : #
Volumetric concentration ratio : # Conc. ratio for HMWC : #
Other : #

Tests and analyses : #

Remarks and description of test.

Date	Initials	
Element		
Product		

Time, H:M:S	Accum. Time, H:M:S	Prefilter pres. In	Prefilter pres. out	Prefilter, pres. Drop	Vol, tank	PermVol	DiaVol	FFLOW	PFLOW	DFLOW	Flux (calculated)	Temperature, Deg C	pH feed	pH concentrate	pH permeate	Pressure, feed	Pressure, concentrate	Pressure drop	Conductivity, feed	Conductivity, conc.	Conductivity, perm.	TS Feed	TS Concentrate	TS permeate	Feed	Concentrate	Permeate	Vol. Conc. Ratio	Sample, marking
		bar	bar	bar	liter	liter	liter	lph	lph	liter	lmh	C	pH	pH	pH	Bar	bar	bar	my-S	my-S	my-S	Brix	Brix	Brix	color	color	color	VCR	

Product / Process	Carrageenan
End user	Industry
Characteristics	Viscous. gelling agent. Food product
Objective	Water removal
Process/waste/drinking water	Food process
Price of product	medium
Established processes	yes
Competing processes	Evaporation and alcohol precipitation
Competing membrane systems	P&F
Use of permeate/concentrate	sewer / product
Production process (outline)	Alkaline extraction at very high temperature
Peculiarities	Do not tolerate pH below 7. Very sensitive to oxidizers Generally VERY viscous. However, it can gel and/or create viscosity.
Pretreatment	Filtration
Membrane choice	10K PSO or PVDF. In reality 50 K will work too.
Element choice	Dairy. 50 mil or higher. D or P. DE construction
TDS	Feed typically 0,5 to 1% Concentrate rarely above 4%
Operating parameters	minimum 85 deg C Pressure drop at least 1 bar per element
Cross flow	Varies a lot due according to viscosity
Cleaning	Low pH High pH hypochlorite
Plant design parameters	Multistage recycle Feed line pressure <3 bar Max. 3 elements per housing. 2 is recommended All stainless steel. Sanitary food grade design
Misc.	Few plants world wide. Technically interesting. Commercially ????
Flux curve	
rejection curve	

Product / Process	Removal of humic and fulvic acid from surface water
End user	Municipality
Characteristics	ice cold clear water
Objective	Reduce content of color and microorganisms
Process/waste/drinking water	drinking
Price of product	low
Established processes	Yes, in Norway
Competing processes	Flocculation
Competing membrane systems	Tubular for small systems Fibers for larger systems
Use of permeate/concentrate	Product / discharge to lake or river
Production process (outline)	Straight forward UF. 90% recovery
Peculiarities	Operating temperature can be below 1 deg C
Pretreatment	Sand filter or cartridge filter
Membrane choice	CA or RC
Element choice	8040F-30D
TDS	negligible
Operating parameters	Pressure max. 6 bar pH and temperature as is
Cross flow	Relatively low. <0,5 bar per element
Cleaning	Hypochlorite Phosphate & citrate
Plant design parameters	Single pass or single stage recirculation Non-sanitary design. PVC and polyester is used a lot
Misc.	March 2001 almost exclusively used in Norway
Flux curve	
rejection curve	

Product / Process	Recovery of dilute milk
End user	Dairy (industry)
Characteristics	High BOD product. Bacteriologically very active
Objective	Recover TDS
Process/waste/drinking water	waste water
Price of product	low
Established processes	no
Competing processes	evaporation
Competing membrane systems	None
Use of permeate/concentrate	sewer / product for cattle fodder
Production process (outline)	The water comes by flushing of tanks and pipes before cleaning.
Peculiarities	Contains a high content of fat on TS. The micelles are often damaged. The raw material not well defined. Watch out for yoghurt - with fruit
Pretreatment	Centrifuge (ideal) or candle filters
Membrane choice	SF
Element choice	8038C-50D or 3838C-50D
TDS	~1% in feed. Final 12%
Operating parameters	Pressure up to 35 bar 10 deg C
Cross flow	High. Typically 18 m3/h per 8" element
Cleaning	Highly alkaline with extra detergent. As high temperature as possible.
Plant design parameters	Multistage recycle. Dairy pumps Clamp connections All dairy style.
Misc.	Cleaning is very difficult. Plant operation very tricky
Flux curve	
rejection curve	

Product / Process	Polyester dyehouse effluent
End user	Industry
Characteristics	Hot, colored
Objective	Recovery water and concentrate TS
Process/waste/drinking water	waste water becoming process water
Price of product	low
Established processes	no
Competing processes	Maybe evaporation
Competing membrane systems	none
Use of permeate/concentrate	use in dye house / sewer
Production process (outline)	Dyeing of polyester yarn with azo dyes
Peculiarities	Contains a tri-mer of phthalate and ethylene glycol
Pretreatment	Cooling from boiling point to 50 - 70 deg C
Membrane choice	SF
Element choice	8040C-DE
TDS	low
Operating parameters	feed pressure below 20 bar temperature not established. from 40 to 90 deg C
Cross flow	High. 0,7 bar per 8040C-DE element
Cleaning	Alkaline detergent
Plant design parameters	Multistage recycle. 90% recovery. several stages. Non-sanitary, all stainless steel
Misc.	Nobody want s such a system. High water prices or lack of water can force them to buy one.
Flux curve	
rejection curve	

Product / Process	Lignosulfonate recovery
End user	Industry
Characteristics	Hot, colored effluent with highly water soluble solutes
Objective	Purify lignosulfonate Either as a product in itself or as an intermediate for vanillin production
Process/waste/drinking water	process
Price of product	medium / high
Established processes	Yes
Competing processes	none
Competing membrane systems	tubular
Use of permeate/concentrate	several / spray drying of alkaline oxidation
Production process (outline)	Acid cooking of wood chips at pH around 1 for 10 hours, at 165 deg C
Peculiarities	Very large volume. Very wide span of MW Extraordinarily water soluble. May contain pitch or rosin (bad news!)
Pretreatment	Filter
Membrane choice	5 to 10K PSO
Element choice	P&F, Tubular. (spirals are rarely used)
TDS	12% in feed. 30 % in concentrate
Operating parameters	5 - 10 bar pressure.
Cross flow	High. Preferably 1 bar per element
Cleaning	Alkaline detergent
Plant design parameters	non-sanitary is fully adequate. Industrial pumps and control systems mandatory
Misc.	Very few systems
Flux curve	
rejection curve	

Product / Process	Low alcohol beer
End user	Industry
Characteristics	High quality beverage. Clear. cold
Objective	Reduce alcohol content from 4% to 0,5%
Process/waste/drinking water	Process
Price of product	High
Established processes	yes
Competing processes	evaporation
Competing membrane systems	none
Use of permeate/concentrate	sewer / product
Production process (outline)	Beer brewing is known.
Peculiarities	Beer is very complex. Membranes shall permeate alcohol and reject aroma compounds of almost the same MW Only diafiltration
Pretreatment	safety filter
Membrane choice	AF
Element choice	AF8038C-30D
TDS	4 - 5%
Operating parameters	4 deg C pressure 25 - 35 bar
Cross flow	high. 1 bar per element
Cleaning	The full Monty
Plant design parameters	Sanitary and food grade is compulsory. Batch system! Batch diafiltration
Misc.	
Flux curve	
rejection curve	

Product / Process	Egg white concentration
End user	industry
Characteristics	somewhat viscous food product
Objective	Concentrate prior to spray drying
Process/waste/drinking water	process
Price of product	Very high
Established processes	yes
Competing processes	none
Competing membrane systems	P&F
Use of permeate/concentrate	sewer / product
Production process (outline)	cracking of egg, separation of white and yolk
Peculiarities	highly shear sensitive contains chalaza RO and fermentation done simultaneously
Pretreatment	filtration at low pressure drop
Membrane choice	AF
Element choice	3840C-50D
TDS	Feed 12%. Conc 20%
Operating parameters	20 deg C
Cross flow	1 bar per element
Cleaning	The full Monty
Plant design parameters	Mostly batch. Low shear pumps and valves Fully sanitary
Misc.	Small and expensive systems. Payback time in weeks rather than months
Flux curve	
rejection curve	

Product / Process	
End user	
Characteristics	
Objective	
Process/waste/drinking water	
Price of product	
Established processes	
Competing processes	
Competing membrane systems	
Use of permeate/concentrate	
Production process (outline)	
Peculiarities	
Pretreatment	
Membrane choice	
Element choice	
TDS	
Operating parameters	
Cross flow	
Cleaning	
Plant design parameters	
Misc.	
Flux curve	
rejection curve	