

## Comparison of commercial membranes in nanofiltration of sweet whey

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**Abstract** – The performance of commercial nanofiltration membranes was studied during concentration and demineralization of sweet whey. The tested membranes were Desal-5 DK, NF45 and Koch SR1. Two different generations of the NF45 membranes were studied. Concentration of whey was made both with a flat-sheet module and in a pilot plant equipped with one spiral wound element. The new generation of the NF45 membrane has comparable retention characteristics with the Desal-5 DK. The Koch SR1 membrane has lower salt and lactose retention than the Desal-5 DK and similar retention characteristics to the older generation of the NF45, which is no longer commercially available. When using the new generation of the NF45 membrane one can expect lower permeate flux and higher retention of salts and lactose than before with the older generation of the NF45 membrane. The highest permeate fluxes of the tested membranes were reached with the Koch SR1 membrane. Concentration of sweet whey with a flat-sheet module and a spiral wound element resulted in similar concentrate composition. Therefore, the flat-sheet module may be used when information on product composition is being investigated. The permeate flux was higher with the flat-sheet module than with the spiral wound element. Thus, either the spiral wound module is more prone to concentration polarization than the flat-sheet module or one can assume that the whole membrane area of the spiral wound module is not used effectively.

**Nanofiltration / spiral wound module / flat-sheet module / sweet whey / concentration**

**Résumé** – **Comparaison de membranes commerciales pour la nanofiltration du lactosérum doux.** Les membranes commerciales de nanofiltration ont été étudiées pour la concentration et la déminéralisation du lactosérum doux. Les membranes testées sont Desal-5 DK, NF45 et Koch SR1. Deux générations de membranes NF45 ont aussi été étudiées. La concentration du lactosérum doux a été effectuée avec un module plat et un pilote équipé d'un élément spiral. La nouvelle génération de

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membranes NF45 a les mêmes caractéristiques de rétention que les membranes Desal-5 DK. Les membranes Koch SR1 ont une rétention en sels et lactose plus faible que les membranes Desal-5 DK et ont les mêmes caractéristiques de rétention que l'ancienne génération de membranes NF45. L'ancienne génération de membranes NF45 n'est plus disponible dans le commerce. L'utilisation des nouvelles membranes NF45 conduit à un flux de perméat réduit et à de meilleures rétentions en sels et lactose en comparaison avec la précédente génération. Les flux de perméats maximaux sont obtenus en utilisant les membranes Koch SR1. Le lactosérum doux concentré avec le module plat a la même composition que celui obtenu avec le module à élément spiral. Par conséquent, le module plat devrait être utilisé lorsque la composition du produit est l'information désirée. Le flux de perméat obtenu avec le module plat est supérieur à celui obtenu avec le module à élément spiral. Le module à élément spiral serait donc plus facilement sujet à une polarisation de concentration que le module plat, à moins que toute la surface de la membrane dans le module à élément spiral ne soit pas utilisée de façon optimale.

### Nanofiltration / module à élément spiral / module plat / lactosérum doux / concentration

#### SYMBOLS AND ABBREVIATIONS

|                     |  |
|---------------------|--|
| $FR_{PWF}$          | Flux reduction of pure water flux, %                                       |
| $Loss$              | Loss of one species, %   |
| $m_{i,concentrate}$ | Mass of one component in the concentrate, g                                |
| $m_{i,feed}$        | Mass of one component in the feed, g                                       |
| $PWF_a$             | Pure water flux after sweet whey filtration, $L \cdot m^{-2} \cdot h^{-1}$ |
| $PWF_b$             | Pure water flux of the clean membrane, $L \cdot m^{-2} \cdot h^{-1}$       |
| $V_{feed}$          | Volume of the feed, $m^3$  |
| $V_{concentrate}$   | Volume of the concentrate, $m^3$   |
| $CIP$               | Cleaning in place  |
| $IEP$               | Isoelectric point  |
| $VCR$               | Volume concentration ratio   |

## 1. INTRODUCTION

In the dairy industry pressure-driven membrane processes are widely used for concentrating different components from dairy fluids, e.g. in producing whey protein concentrates [15, 22]. Some membrane processes improve product manufacturing technology, e.g. ultrafiltration in the Feta cheese process [4, 25], and the others

reduce effluent streams in dairying, e.g. water, chemical and brine recycling [6, 7, 25].

Nanofiltration is one of the pressure-driven membrane processes and the speciality of it is low retention towards monovalent ions. The separation mechanisms of nanofiltration membranes consist of steric and electrical effects. Nanofiltration of a multicomponent solution is a very complex process with numerous interactions. Nanofiltration membranes do not have real visible pores, but they have some free volume depending on their openness and structure [20]. Košutić et al. [17] defined a pore in a nanofiltration membrane as a polymer material-free void space through which fluids can be transported under a driving force, i.e. pressure. In some cases pores might have been seen in atomic force microscopy (AFM) [1]. Many commercial nanofiltration membranes are thin-film composite membranes that have an active layer formed from an aromatic cross-linked polyamide. Most membranes are negatively charged at neutral pH [23, 31].

The most important principle concerning electrical effects is the Donnan equilibrium principle. The Donnan equilibrium principle requires an equal electrochemical potential on both sides of a membrane and can thus increase the permeation of a less charged component when retaining a more charged one [3]. Multivalent ions and

disaccharides are rejected by nanofiltration membranes. In other words, the separation characteristics of nanofiltration are between reverse osmosis and ultrafiltration [14]. It should be noted that because membranes have very different separation characteristics, the point of differentiation between the various systems is seldom distinct. Thus, the limits between different pressure-driven membrane processes are vague [21]. The filtration pressures used in nanofiltration are usually between 0.6 and 4 MPa; when in reverse osmosis up to 12 MPa are used, and in ultrafiltration pressures are under 1 MPa. Nanofiltration has replaced many old reverse osmosis plants and in total 150 000–200 000 m<sup>2</sup> of nanofiltration area is installed in the dairy industry all over the world [13].

Concentration and demineralization of whey has been one important application of membrane technologies in the dairy industry for the last fifteen years [8]. Whey is a by-product of little value as a result of its high salt content and should be processed as easily and economically as possible. Characteristics of a good membrane include a high permeate flux, low retention of salts and low fouling tendency. Anyhow, pre-treatment of whey is recommended to prevent long-term fouling. The most commonly used pre-treatments are fat and casein fines separation, pasteurization and pH adjustment [24]. In the nanofiltration process a high retention of lactose is desired from the economical and environmental point of view. As understood, this latter goal is in contradiction with the desire to achieve low retention of salts.

The total solids content of whey is 5.0–6.0%, of which approximately 80% is lactose and 10–15% are proteins [19, 30]. Partial demineralization of whey by nanofiltration increases its value as it makes whey concentrate more suitable for human food additive or animal feed. In nanofiltration, whey is concentrated to 15–25% total solids and simultaneously the

total mineral content is reduced to 40–50%. The loss of lactose is 1–5% [16, 27, 29]. It is noted also that the composition of the whey concentrate varies widely depending on the process used and the specification limits set. The higher the total solids content, the greater the risk of problems occurring in the nanofiltration process. Particularly the deposition of calcium phosphate is a commonly recognized problem.

The aim of this study was to compare commercial nanofiltration membranes in sweet whey concentration and demineralization. In addition, flat-sheet module runs were compared with spiral wound module runs.

## 2. MATERIALS AND METHODS

### 2.1. Membranes

Three different nanofiltration membranes were used: Desal-5 DK, NF45 and Koch SR1. According to the manufacturer of the NF45 membrane the manufacturing process of the membranes changed in the spring of 2000 and this has caused changes in the characteristics of the NF45 membrane. However, the NF45 membrane is still sold under the same brand name. Both generations of the NF45 membranes were studied, old NF45 and new NF45, respectively. The old NF45 membrane is no longer commercially available. The spiral wound elements used in the test runs were suitable for food processing applications requiring stringent sanitary procedures. Some properties of the membranes used are presented in Table I.

### 2.2. Sweet whey

The test whey was sieved, separated and pasteurized sweet whey. Its pH was adjusted to 6.0 by hydrochloric acid. The whey was taken from a feed line of an industrial scale whey NF processing system.

**Table I.** Description of the nanofiltration membranes used in this study.

|                                  | Desal-5 DK    | NF45                      | Koch SR1                   |
|----------------------------------|---------------|---------------------------|----------------------------|
| Manufacturer                     | Osmonics, USA | Dow Chemical Company, USA | Koch Membrane Systems, USA |
| Material                         | Polyamide     | Polyamide                 | Polyamide                  |
| Model                            | DK3840C-1097  | 3838/30-FF                | TFC-3838SR1-N1             |
| Max. operating pressure, MPa     | 4.14          | 5.48                      | 4.10                       |
| Max. operating temperature, °C   | 50            | 45                        | 50                         |
| pH-range in continuous operation | 3–10          | 2–10                      | 4–10                       |

**Table II.** The average composition of the sweet whey used (10 batches).

|                                 | Sweet whey  |
|---------------------------------|-------------|
| pH                              | 6.00 ± 0.01 |
| Conductivity, S·m <sup>-1</sup> | 0.57 ± 0.02 |
| Solids, %                       | 5.16 ± 0.02 |
| Ash, %                          | 0.49 ± 0.01 |
| Sodium, mg·kg <sup>-1</sup>     | 349 ± 8     |
| Potassium, mg·kg <sup>-1</sup>  | 1450 ± 28   |
| Calcium, mg·kg <sup>-1</sup>    | 358 ± 24    |
| Magnesium, mg·kg <sup>-1</sup>  | 69 ± 8      |
| Phosphorus, mg·kg <sup>-1</sup> | 346 ± 16    |
| Chloride, mg·kg <sup>-1</sup>   | 1080 ± 18   |
| Lactose, g·L <sup>-1</sup>      | 35.4 ± 6.4  |
| Protein, %                      | 0.64 ± 0.01 |

The average composition of the sweet whey used is shown in Table II.

The osmotic pressure of sweet whey calculated with the van't Hoff's equation [2] by taking into account ash and lactose is 0.5 MPa and the calculated viscosity of sweet whey at 20 °C is 1.2 MPa and at 15 °C 1.4 MPa [26].

### 2.3. Nanofiltration equipment

#### 2.3.1. Flat-sheet module

The laboratory experiments were carried out with the LabStak M20-0.72 (Danish Separation Systems AS, Nakskov, Denmark). The total membrane area was

0.216 m<sup>2</sup>, i.e. 12 pieces of membrane, each of an area of 0.018 m<sup>2</sup>. All membranes were tested in parallel experiencing the same feed, although there is a possibility of placing membranes in series and testing them stagewise.

During the test runs permeate was collected in the filtrate tank and the retentate was returned to the feed tank. The filtrate tank was placed on a scale to measure the accumulating permeate. During the test runs the temperature of the whey was held constant by cooling down the circulating retentate and the feed with a glycol-water stream cryostat (Lauda WK4600, Lauda DR.R. Wobser GmbH., Lauda-Königshofen, Germany).

### 2.3.2. Pilot module setup equipped with one spiral wound element

The spiral wound module tests were carried out with a pilot setup (Valmet Flootek AB, Raisio, Finland) equipped with one spiral wound module. The measurement system was principally similar to the one used in the flat-sheet module experiments. The volume of permeate was measured and the retentate was circulated back to the feed tank. The pilot setup used is presented in Figure 1.

The membrane area was 7.4 m<sup>2</sup> with the Desal-5 DK and the NF45 elements and 6.5 m<sup>2</sup> with the Koch SR1 element.

### 2.4. Nanofiltration experiments

The experiments with the flat-sheet module were done at two different pressures, 1.5 MPa and 2.0 MPa, and at two temperatures, 15 °C and 20 °C, respectively. The feed flow of whey was 0.35 m<sup>3</sup>·h<sup>-1</sup>. The whey was concentrated to the volume concentration ratio (VCR) 4. Before the test, the filtration membranes went through a one-stage cleaning-in-place procedure. A 30 min basic wash (P3-Ultrasil 10, Henkel-Ecolab, Düsseldorf, Germany) at pH 11 was made at 1.5 MPa

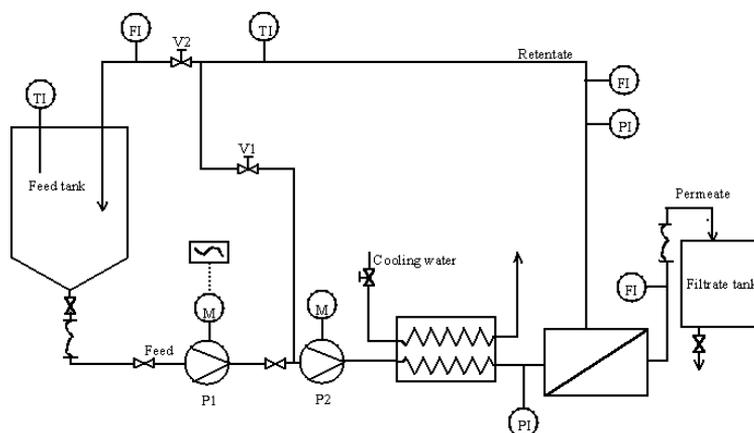
pressure. The equipment was then flushed with water until a pH value between 6 and 8 was reached and then the pure water flux was measured. All water used in the filtration experiments was tap water. After whey filtration the membranes were flushed with water without any applied pressure for 5 min (feed flow 0.35 m<sup>3</sup>·h<sup>-1</sup>) and then the pure water flux was measured.

The pilot scale experiments begun with a cleaning-in-place (CIP) procedure at 0.5 MPa and 42 °C. The CIP procedure mimics an industrial scale CIP. The procedure was as follows:

1. 20 min acidic wash at pH 2;
2. 30 min basic wash at pH 11;
3. 10 min disinfection with a sodium hexametaphosphate-based product.

There was a water flushing after every stage of the CIP procedure. The water flushing continued until a pH value between 6 and 8 was reached. The pure water flux was measured at 2.0 MPa and 20 °C after the CIP procedure.

The pilot scale nanofiltration experiments were made at 2.0 MPa and at 20 °C. Every spiral wound module was tested twice. 0.5 m<sup>3</sup> of sweet whey was concentrated to a volume of 0.1 m<sup>3</sup>, that is, a VCR of 5. The feed flow to the element was



**Figure 1.** The pilot setup equipped with one spiral wound module used in nanofiltration experiments.

5.4 m<sup>3</sup>·h<sup>-1</sup>. During the whey filtration permeate flux was measured and samples were taken from the cumulative permeate and the retentate. After whey filtration, the membrane element was flushed with water for 10 min and the pure water flux was measured. The membrane element was cleaned following the *CIP* procedure.

The reproducibility of the filtration tests was studied with replicate runs both with the spiral wound and the flat-sheet filtration tests. The replicate runs gave the same permeate fluxes versus filtration time. The minor differences in permeate flux values between the replicate runs are due to the inaccuracy in the volumetric measurement. The measurements were thus considered reproducible.

### 2.5. Methods of analysis

pH was measured with a Mettler Delta 320 pH-meter (Mettler-Toledo Ltd., Halstead, Great Britain) and conductivity with a WTW LF320 conductivity meter (WTW GmbH, Weilheim, Germany). Total solids and ash were analyzed according to the IDF methods [9, 11]. Lactose was detected with the Lactose/D-Galactose Enzymatic BioAnalysis (Cat. No. 176306, Boehringer, Mannheim, Germany) with UV-VIS at the wavelength 340 nm. Minerals (Na<sup>+</sup>, K<sup>+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>) were analyzed with the AAS method. Chloride was titrated according to the IDF method [10]. Phosphorus was detected with a MIRA S autoanalyzer (Roche Diagnostics, F. Hoffmann-La Roche Ltd, Basel, Switzerland) by a method based on the phosphomolybdate reaction. Protein was analyzed according to the IDF method [12].

### 2.6. Calculation of the parameters used to evaluate the filtration performance

The volume concentration ratio measures the concentration degree of sweet whey.

$$VCR = \frac{V_{\text{feed}}}{V_{\text{concentrate}}} \quad (1)$$

where *VCR* is the volume concentration ratio,  $V_{\text{feed}}$  (m<sup>3</sup>) the volume of the feed in the beginning and  $V_{\text{concentrate}}$  (m<sup>3</sup>) the volume of the concentrate (retentate) in the end.

The losses of various components were calculated by comparing the mass of the substance in the concentrate with that of the feed as follows:

$$Loss = \left( 1 - \frac{m_{i,\text{concentrate}}}{m_{i,\text{feed}}} \right) 100\% \quad (2)$$

where *Loss* is the loss of one component (%),  $m_{i,\text{concentrate}}$  the mass of one component in the concentrate (retentate) and  $m_{i,\text{feed}}$  the mass of that component in the feed. The loss of lactose is by way of exception calculated by comparing its mass in both the permeate and feed. This is common practice in industry although concentrated sweet whey is the product in this case.

The flux reduction of the pure water flux was calculated by comparing pure water flux before and after the filtration as follows:

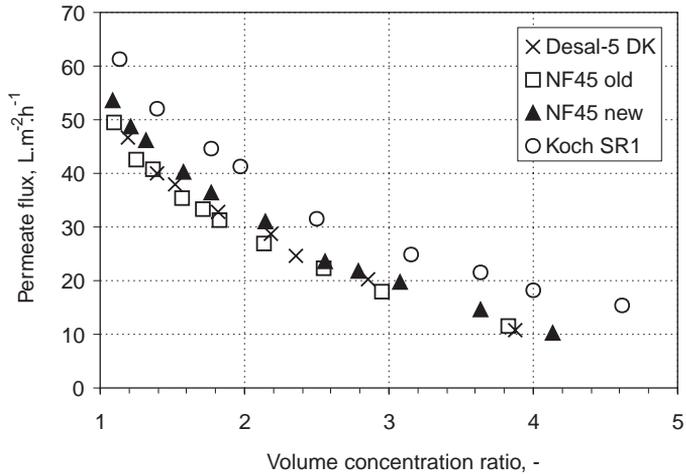
$$FR_{\text{PWF}} = \frac{PWF_b - PWF_a}{PWF_b} 100\% \quad (3)$$

where  $FR_{\text{PWF}}$  is the reduction in pure water flux,  $PWF_b$  (L·m<sup>-2</sup>·h<sup>-1</sup>) the pure water flux of the clean membrane and  $PWF_a$  (L·m<sup>-2</sup>·h<sup>-1</sup>) the pure water flux after sweet whey filtration.

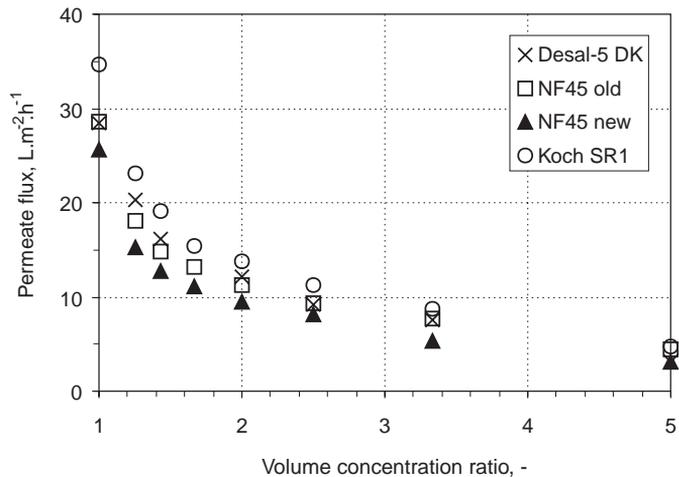
## 3. RESULTS AND DISCUSSION

Permeate fluxes during whey filtration were measured in order to establish differences in the capacities of the membranes. No significant differences in permeate fluxes were detected between the Desal-5 and the NF45 membranes in the flat-sheet tests. The Koch SR1 membrane gave on

**Figure 2.** Permeate fluxes in sweet whey concentration with a laboratory scale flat-sheet module at 2.0 MPa and 20 °C. The membrane area was 0.216 m<sup>2</sup> and the feed flow of whey was 0.35 m<sup>3</sup>·h<sup>-1</sup>.



**Figure 3.** Permeate fluxes in sweet whey concentration with a spiral wound element at 2.0 MPa and 20 °C. The membrane area was 7.4 m<sup>2</sup> with the Desal-5 DK and the NF45 membrane elements and 6.5 m<sup>2</sup> with the Koch SR1 membrane element. The feed flow to the element was 5.4 m<sup>3</sup>·h<sup>-1</sup>.



average 10 L·m<sup>-2</sup>·h<sup>-1</sup> higher permeate fluxes than the other membranes. The permeate fluxes in the flat-sheet tests are shown in Figure 2.

The LabStak M20 flat-sheet module was also used by Vasiljevic and Jelen [29] in nanofiltration of Cottage cheese whey with a membrane area of 0.036 m<sup>2</sup>. A permeate flux of 45 L·m<sup>-2</sup>·h<sup>-1</sup> at 2.0 MPa and 20 °C was obtained at VCR 1 with the NF45PE membrane. According to Figure 2 the fluxes of the Desal-5 and the NF45 membranes are in agreement with the measurements of Vasiljevic and Jelen

when taking into account that fluxes with acid whey, i.e. Cottage cheese whey, are usually 10–20% lower than with sweet whey.

In the pilot plant spiral wound experiments the highest permeate fluxes were also reached with the Koch SR1 membrane. The new NF45 membrane gave slightly lower fluxes than the old NF45 membrane. At the beginning of whey concentration the permeate flux was 35 L·m<sup>-2</sup>·h<sup>-1</sup> with the Koch SR1 membrane and 26 L·m<sup>-2</sup>·h<sup>-1</sup> with the new NF45 membrane. At VCR = 5 the permeate flux was 5 L·m<sup>-2</sup>·h<sup>-1</sup> with the

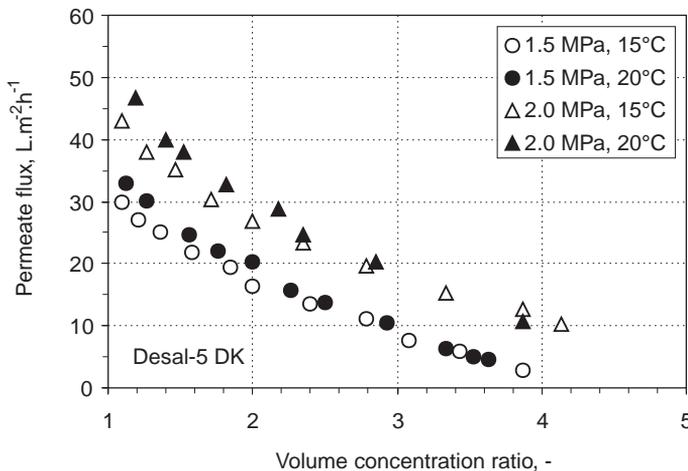
Koch SR 1 membrane and  $3 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$  with the new NF45 membrane. The permeate fluxes in the pilot scale experiments are shown in Figure 3.

The permeate fluxes were higher with the flat-sheet module than with the spiral wound module as can be seen from Figures 2 and 3. Although the cross-flow conditions in the flat-sheet module are different from the spiral wound module, this could indicate that there are dead zones in the spiral wound modules. The inefficient use of membrane area in spiral wound modules has been shown by van Gauwbergen and Baeyens [28]. On the other hand, in spiral wound tests the permeate flux decreases rapidly at the beginning so that when the *VCR* is larger than 2 the plot of the permeate flux versus the *VCR* looks almost linear (Fig. 3). The decrease in the permeate flux results presumably from concentration polarization. The concentration polarization layer on the membrane surface grows and the osmotic pressure increases as the concentration of whey advances. The approximate osmotic pressures at *VCR* 5 in sweet whey concentrate and permeate are 1.4 MPa and 0.8 MPa, respectively, calculated with the van't Hoff's equation [2] by taking into account ash and lactose. Thus,

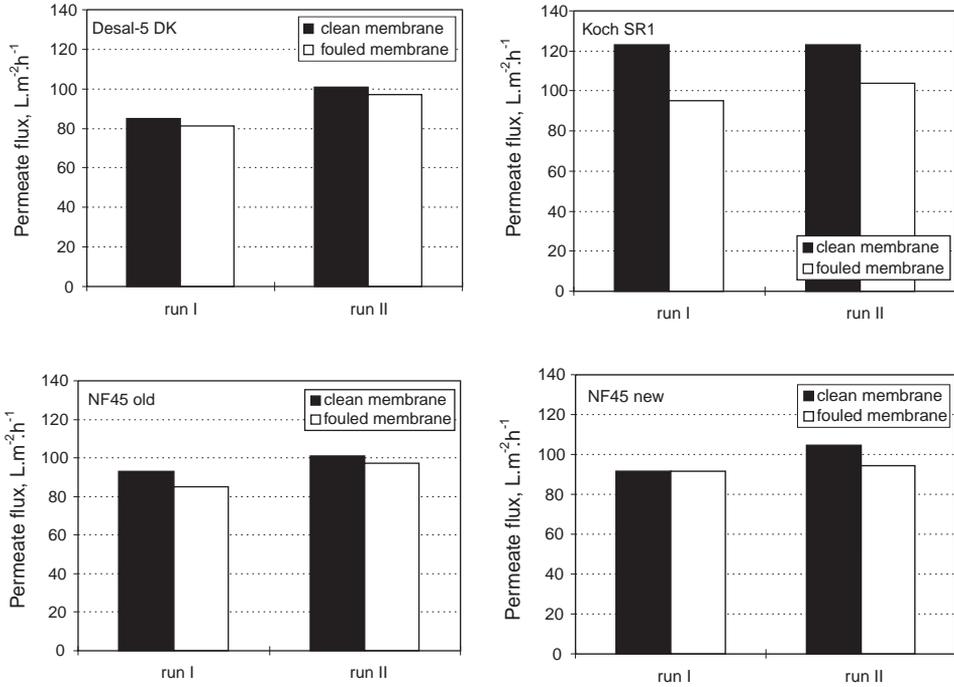
the transmembrane osmotic pressure is approximately 0.6 MPa. These estimates are presumably lower than the real osmotic pressure resulting from its dependence on the pH and the ionic strength of this protein containing solution [2].

The influence of filtration pressure and temperature was studied in the flat-sheet tests. Figure 4 shows the impact of pressure and temperature on sweet whey nanofiltration with the Desal-5 membrane. According to Figure 4, the applied pressure has a major impact on permeate flux. Permeate fluxes are also higher at 20 °C than at 15 °C when *VCR* is smaller than 2. At greater *VCR* values when the pressure is held constant, the influence of a 5 °C temperature rise is inconsequential. This is probably viscosity changes due to relative solids increase being greater than the effect of temperature change on whey. The influence of temperature on permeate fluxes should be further studied to find out the dependence between viscosity and critical flux.

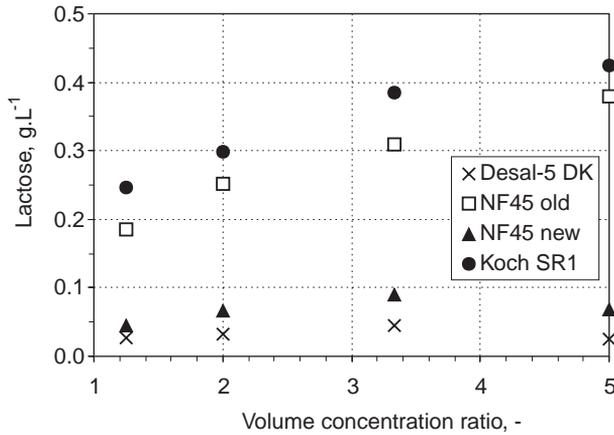
The pure water flux at 2.0 MPa and 20 °C for a clean membrane was measured after the *CIP* procedure and for a fouled membrane after water flushing. Pure water fluxes are shown in Figure 5. The Koch SR1



**Figure 4.** Influence of pressure and temperature on permeate fluxes in sweet whey nanofiltration with a laboratory scale flat-sheet module. The membrane area was  $0.216 \text{ m}^2$  and the feed flow of whey was  $0.35 \text{ m}^3\cdot\text{h}^{-1}$ .



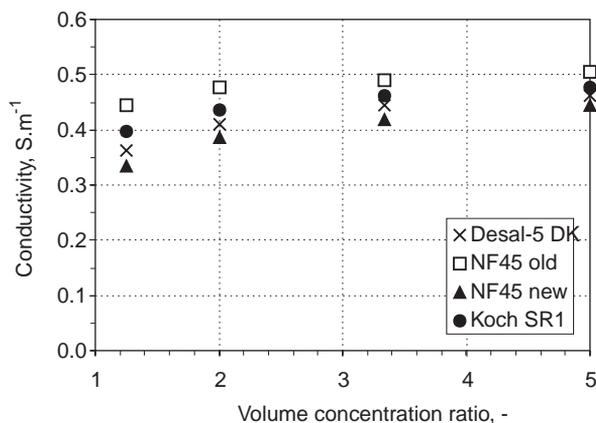
**Figure 5.** Pure water fluxes measured twice in spiral wound modules at 2.0 MPa and 20 °C, for various membranes, clean and fouled. A CIP cleaning was done before each run. Every spiral wound module was new prior to run I.



**Figure 6.** Lactose contents in cumulative permeates in sweet whey concentration with spiral wound modules at 2.0 MPa and 20 °C.

membrane gives the highest pure water flux as a clean membrane. However, the reduction of the pure water flux is over 15% for the Koch SR1 compared to <10% for the

other tested membranes. Further study is necessary to establish whether irreversible fouling weakens the high capacity of the Koch SR1 membrane in the long run.



**Figure 7.** Conductivities in cumulative permeates in sweet whey concentration with spiral wound modules at 2.0 MPa and 20 °C.

Fouling of the Desal-5 and the NF45 membranes is minor. In fact, these membranes when treated twice with the *CIP* procedure give higher pure water fluxes than when treated only once. A similar behavior in cleaning was observed by Mänttari and Nyström [18]. This seems to indicate that the membranes did not foul irreversibly. These results are compatible with the conclusions of reversible fouling by Jeantet et al. [13].

### 3.1. Loss of lactose and ash

Performance based on permeation of lactose and salts divided the tested nanofiltration membranes into two groups. The lactose content in the cumulative permeate in the pilot scale tests is shown in Figure 6. The Desal-5 and the new NF45 membranes have significantly lower permeation of lactose than the other two membranes. The Koch SR1 membrane gives the highest lactose concentration in the permeate compared to the other membranes.

The flat-sheet nanofiltration tests did not show any difference in lactose permeation between the two generations of the NF45 membrane. The lactose content of the permeate was 0.15 g·L<sup>-1</sup> at VCR 4 for both NF45 generations. However, the Desal-5 membrane gave the lowest lactose concen-

tration in the permeate, <0.1 g·L<sup>-1</sup>, and the Koch SR1 membrane the highest, 0.3 g·L<sup>-1</sup>, at VCR 4.

The loss of lactose in the spiral wound module tests with Desal-5 and new NF45 membranes is low, less than 0.1%. The higher lactose contents in the permeates of the Koch SR1 and the old NF45 membranes indicate higher losses of 1.0% with these looser membranes when concentrating to VCR 5.

The conductivity of the cumulative permeate is shown in Figure 7. The conductivity of the permeate increases as the whey becomes more concentrated. Thus, according to the Donnan equilibrium principle the permeation of salts increases as the VCR rises.

Loss of minerals, phosphorus and chloride in the spiral wound module tests at VCR 5 are shown in Table III. The permeation of specified anions and cations (Tab. III) confirms the similarity of retention characteristics between the Koch SR1 and the old NF45 membranes and between the Desal-5 and the new NF45 membranes, respectively. All the membranes gave high passage of monovalent chloride ion. Thus, the chloride concentration in the sweet whey concentrate (i.e. retentate at VCR 5) was lower than in the original sweet whey. Membranes differed with respect to losses

**Table III.** Loss of some minerals, phosphorus and chloride in sweet whey concentration at VCR 5 in the pilot scale experiments with one spiral wound element.

|            | Loss, %    |          |          |          |
|------------|------------|----------|----------|----------|
|            | Desal-5 DK | NF45 old | NF45 new | Koch SR1 |
| Sodium     | 64         | 70       | 61       | 68       |
| Potassium  | 63         | 68       | 61       | 68       |
| Calcium    | 23         | 26       | 29       | 29       |
| Magnesium  | 21         | 24       | 26       | 24       |
| Phosphorus | 27         | 42       | 27       | 45       |
| Chloride   | 84         | 85       | 83       | 82       |

**Table IV.** The composition of sweet whey concentrate with different spiral wound membranes.

|                                 | Desal-5 DK | NF45 old | NF45 new | Koch SR1 |
|---------------------------------|------------|----------|----------|----------|
| pH                              | 5.7        | 5.7      | 5.8      | 5.8      |
| Conductivity, S·m <sup>-1</sup> | 0.67       | 0.59     | 0.67     | 0.58     |
| Solids, %                       | 17.7       | 17.3     | 17.9     | 17.8     |
| Ash, %                          | 1.08       | 0.94     | 1.09     | 0.96     |
| Lactose, g·L <sup>-1</sup>      | 128        | 122      | 131      | 136      |
| Protein, %                      | 2.07       | 2.04     | 2.07     | 2.07     |

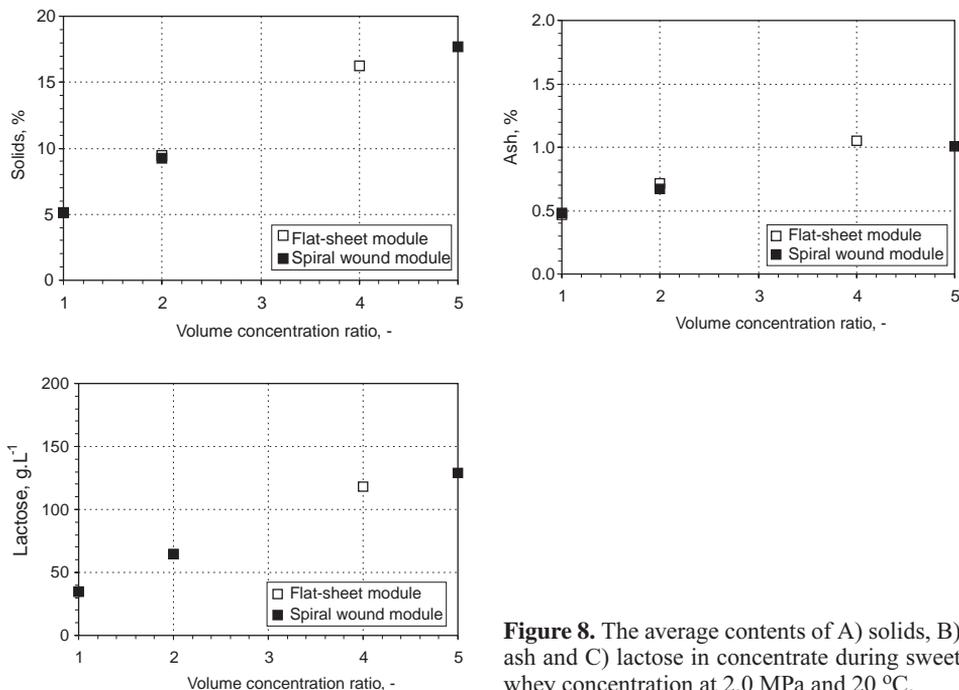
of multivalent phosphorus. Only 27% of the phosphorus passed the tighter membranes, the Desal-5 and the new NF45. The looser membranes, the Koch SR1 and the old NF45, gave passage to over 40% of the phosphorus present in sweet whey.

The permeation of salts could be enhanced by adjusting the pH of sweet whey to the isoelectric point (*IEP*) of the membrane [5]. This would most certainly also enhance the permeation of lactose. The *IEP* values of commercial membranes are not commonly available. In addition, whey proteins are prone to precipitation when pH drops under 5.8, and calcium phosphate precipitates when pH rises above 6.2. A drastic adjustment of pH could, therefore, instead of enhancing permeation of salts, lead to heavy fouling of the membrane.

Permeation of salts might be enhanced with diafiltration. However, if diafiltration is also used, more lactose is lost and a longer concentration cycle is needed. From the industrial point of view nanofiltration is a whey concentration method that is used instead of evaporation. The partial demineralization is one of the advantages of nanofiltration. In order to manufacture commercial demineralized whey products, either electrodialysis or ion exchange, or their combination, is needed.

### 3.2. Composition of sweet whey concentrate

The composition of sweet whey concentrates produced by the different membranes is shown in Table IV. The conductivities



**Figure 8.** The average contents of A) solids, B) ash and C) lactose in concentrate during sweet whey concentration at 2.0 MPa and 20 °C.

and ash contents of the sweet whey concentrates from the Desal-5 and the new NF45 membranes are higher than the respective values of the Koch SR1 and the old NF45 membranes. However, the contents of solids and lactose do not show any remarkable differences.

The relatively low value of solids in the concentrate is partly due to the permeation of salts, lactose and nitrogen through the membrane. Nitrogen passes the membrane in the form of urea and this causes the loss in the protein content of the concentrate as the protein analysis is actually a nitrogen analysis [12]. Parts of the solids were not obtained in the concentrate because they were retained in the system.

Figure 8 shows the average contents of solids, lactose and ash in the concentrate of the flat-sheet and the spiral wound module experiments. It is evident that the composition of the sweet whey concentrate remains the same regardless of the type of filtration

module used. Actually, the lactose content of the concentrate was equal at VCR 1 and VCR 2. One could have assumed that differences in cross-flow conditions in different module types would have had an influence on the permeation of components. Anyhow, according to the results shown in Figure 8 these differences have a minor effect on permeation.

#### 4. CONCLUSIONS

The scope of this study was to examine the differences between commercial nanofiltration membranes in sweet whey concentration and demineralization. In addition, the differences between two generations of the NF45 membranes were studied. The manufacturing process and, therefore, the separation characteristics of the NF45 membranes have changed during the year 2000.

In conclusion, one may expect to reach higher capacities in whey processing with the Koch SR1 membrane than with the Desal-5 DK or the NF45 membranes, when the composition of the retentate or permeate is not taken into consideration. However, the reduction of the pure water flux was higher with the Koch SR1 membrane than with the other two membranes. This result may help predict fouling problems in long-term sweet whey concentration.

The membranes studied can be divided into two groups with different retention characteristics. The first group of tighter membranes consists of the Desal-5 DK and the new NF45. The loss of lactose was only 0.1% and the reduction in ash content of the retentate (*VCR* 5) was 53%. The second group of looser membranes consists of the Koch SR1 and the old NF45, which is no longer commercially available. These membranes gave a total loss of lactose of 1.0% and the reduction of the ash content was 58%.

According to the flat-sheet module tests, filtration pressure has a stronger influence on permeate flux than temperature. The permeate flux at 2.0 MPa was  $10 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$  higher than at 1.5 MPa. A temperature change of 5 degrees seemed to have an impact only when the *VCR* was lower than 2, as higher permeate flux values were reached at the higher temperature.

The same composition of sweet whey concentrate was obtained with flat-sheet and spiral wound modules. The difference between these modules is in the permeate flux values. Anyhow, the substantially lower permeate flux values of the spiral wound modules imply that the spiral wound modules are more prone to concentration polarization than the flat-sheet modules. Another assumption is that the whole membrane area is not effectively in use. The flat-sheet module studies gave adequate information on the composition of the sweet whey concentrate. However, the production capacity of a NF system consisting of spiral

wound elements needs to be studied using pilot scale spiral wound module systems.

To conclude, the retention characteristics of the Koch SR1 membrane equal the old generation of the NF45 membrane. The new generation of the NF45 membrane is equivalent to the Desal-5 DK. When displacing the old generation of the NF45 membrane with the new generation, one can expect lower permeate flux and higher retentions of salts and lactose.

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