Manual on Nanofiltration
Introduction
Nanofiltration can be used in drinking water supply for different reasons including heavy metal removal, organic matter removal, calcium and magnesium removal as well as in the treatment of surface water. Nanofiltration is capable of retaining iron and manganese ions as well as organic matter when treating anaerobic groundwater. This treatment technique can also be used to soften water by retaining calcium and magnesium ions thereby reducing water hardness.

Membrane filtration is an advanced treatment process. In this experiment we will introduce the basic principles of nanofiltration and the possibilities for system operation will be demonstrated.

Membrane flux
The driving force for membrane filtration is a pressure difference between the feed water and permeate. This pressure difference results in a flow of water through the membrane according to equation 1.

\[ J = \frac{K_w}{\mu} (TMP - \Delta\pi) \]  

Where:
- \( J \) = flux (m/s)
- \( K_w \) = membrane water permeability (m)
- \( TMP \) = Trans Membrane Pressure (Pa)
- \( \mu \) = absolute viscosity (Pa*s)
- \( \Delta\pi \) = osmotic pressure difference (Pa)

The membrane water permeability \( (K_w) \) is dependent on the size of the pores in the membrane, the porosity of the membrane and the thickness of the membrane. The membrane water permeability is constant over time and independent of the feed water quality.

Trans membrane pressure \( (TMP) \) is the pressure difference between the feed and the permeate side of the membrane, and is calculated as follows:

\[ TMP = \frac{P_f + P_c}{2} - P_p \]  

Where
- \( P_f \) = pressure of feed (Pa)
- \( P_c \) = pressure of concentratie (Pa)
- \( P_p \) = pressure of permeate (Pa) (about zero)

Osmotic pressure is a liquid property dependent on the salt concentration of the liquid, and is calculated from the conductivity:

\[ \Pi = 0.0252 \times EC \]  

\( \Pi \) = osmotic pressure in kPa
\( EC \) = electric conductivity in \( \mu S/cm \) at \( 20^\circ C \)

This formula is only valid for MgSO₄ solutions

Because salts are retained by the membrane, there is an osmotic pressure difference \( (\Delta\pi) \) over the membrane. This osmotic pressure difference is calculated as:
\[ \Delta \pi = \pi_{\text{membrane}} - \pi_{\text{permeate}} \]  

Where:
\( \pi_{\text{membrane}} \) = osmotic pressure at membrane surface at the feed side (Pa)
\( \pi_{\text{permeate}} \) = osmotic pressure of the permeate (Pa)

Finally, the absolute viscosity (\( \mu \)) can be calculated using the following formula:
\[ \mu = \nu \cdot \rho_w = \frac{497 \cdot 10^{-6}}{(42.5 + T)^{1.5}} \cdot 1000 = \frac{497 \cdot 10^{-3}}{(42.5 + T)^{1.5}} \]  

Where:
\( \mu \) = absolute viscosity (N*s/m² or Pa.s)
\( \nu \) = kinematic viscosity (m²/s)
\( \rho_w \) = density of water (kg/m³)
\( T \) = temperature (°C)

**Concentration polarisation**
The membrane used in the experimental setup is a crossflow membrane. This type of membrane both has a water flow through the membrane (which becomes permeate) and a water flow across the membrane. When water flows through the membrane, solutes are retained by the membrane and accumulate at the membrane surface in a so-called “boundary layer”. This is a stagnant water layer of a few micrometer close to the membrane.

The thickness of the boundary layer is depending on the cross flow velocity. The higher the cross flow velocity, the thinner the boundary layer and this will result in a low concentration polarisation. The increased concentration at the membrane wall results in a higher osmotic pressure difference and thus a lower driving pressure and thus a lower flux.
Materials and Methods

Experimental setup
The membrane module is the central part in the installation (Figure 1). This module contains 45 capillary nanofiltration membranes with an inner diameter of 1.5 mm and a length of 1 m. The module is positioned vertically. Feed water is added to the module by an adjustable pump. The flow of the feed \(Q_f\) and permeate \(Q_p\) are measured with an in-line flow meter. The operating pressure is measured at two different places in the system, in the feed flow \(P_f\) and concentrate flow \(P_c\), and can be changed by adjusting the pressure valve in the concentrate pipe. The permeate is at atmospheric pressure. The electrical conductivity and temperature of the feed water (EC\(_f\)) and permeate (EC\(_c\)) are measured. With three-way valves, the feed water source (demiwater or MgSO\(_4\)-containing solution) can be changed. The installation is fully computer-controlled and monitored. Pay special attention to the pump: do not increase the pump speed too fast. The electromotor and the pump are coupled by magnets. This is also a safety measurement! The magnetic transmission can uncouple. You will notice this because the feed pressure and the feedflow will drop. You have to slow down almost to zero speed before the pump and the electromotor are coupled again.

![Figure 1: Schematic drawing of the test installation](image)

Online login procedure

Experiment 1:
The equipment is a scale model of a real reverse osmosis installation:
Such installation are also computer controlled and the installation is regulated by adjusting the feedpump and the concentrate valve. The goal is to obtain a certain flux and a certain cross flow velocity. The flux is important for the production and the cross flow velocity is important for controlling concentration polarisation. Try to obtain the following fluxes and cross flow velocities (using demiwater): Try always to stay below 5 bar!!!

<table>
<thead>
<tr>
<th>Flux l/(m2.h)</th>
<th>Cross flow m/s</th>
<th>Feed pressure bar</th>
<th>Concentrate pressure bar</th>
<th>Recovery %</th>
</tr>
</thead>
</table>
**Experiment 2: determination of the water permeability of the nanofiltration membrane**

In this experiment, the permeate production is measured during 15 minutes, with demineralised water as feed water. The feed pressure is 3 bar and the cross-flow velocity is 1.25 m/s.

Procedure:
- Calculate the flow rate to obtain a crossflow velocity of 1.25 m/s in the membrane module
- Check both 3-way switches; they should take the feed water from the demiwater vessel, and return the concentrate to it.
- Turn on the pump and adjust the pump rotation speed (rounds per minute; rpm) and pressure valve such that a feed pressure (P_f) of 3.0 bar is obtained, and the calculated crossflow velocity is reached.
- Make sure that the pressure valve opening percentage ALWAYS is >10%.
- Wait until the permeate concentration is constant (this can take some minutes)
- Check if the feed pressure is still 3 bars and if the flow rate is as desired. If not, adjust accordingly.
- Start the experiment by measuring conductivity, permeate flow and temperature for every minute during 15 minutes.
- Stop the experiment after 15 minutes of filtration by shutting down the pump.
- With demiwater the osmotic pressure is almost zero and formula 1 can be used to calculate the membrane permeability

**Experiment 3: Filtration of water containing bivalent ions**

In this experiment, use water from the vessel with a MgSO_4·7H_2O concentration of 12 g/l. Again, water is filtered at a pressure of 3 bars during 15 minutes and the cross flow velocity is 1.25 m/s. The only difference of this experiment as compared to experiment 1 is the feed water, so after switching the 3-way valves to the a MgSO_4·7H_2O vessel, the same procedure is followed as in experiment 1.

**Experiment 4: influence of cross flow velocity on system performance**

In this final experiment, once again we use water from the storage vessel with a MgSO_4·7H_2O concentration of 12 g/l. Water is filtered at a pressure of 3 bars during 15 minutes. The cross flow velocity is lowered to 0.35 m/s. The procedure of this experiment is the same as experiment 2, with the only difference that a lower cross flow velocity is used.

At the end of experiment 3, switch the feed of the membrane back to the vessel with demineralized water and then switch the concentrate return back to the demiwater vessel. If you do this the other way around the demiwater vessel will be polluted with MgSO4.

**Results and Discussion**

**Experiment 1**
Fill in the table

**Experiment 2**
1. Calculate the flow rate used to obtain a cross flow velocity of 1.25 m/s
2. Present the experimental results graphically and give comments on the results
3. Calculate the TMP of the experiment
4. Explain why the electrical conductivity of the feed water and the permeate are low
5. Determine the membrane water permeability
6. Explain why the permeability is constant

**Experiment 2**
7. Present the experimental results graphically and include comments on the results
8. Calculate the recovery of the system
9. Calculate the rejection of MgSO₄ based on the EC of feed and permeate
10. Calculate the theoretical osmotic pressure of the feed, concentrate and permeate with the equation given in the hand-out. Include comments on the results.
12. Calculate the concentration polarization (cₓₜ₉).

**Experiment 3**
13. Present the experimental results graphically and give comments on the results.
14. Calculate the recovery of the system
15. Calculate the rejection of MgSO₄ based on the EC
16. Calculate the theoretical osmotic pressure of the feed, concentrate and permeate with the equation given in the hand-out. Include comments on the results.
17. Calculate the concentration polarization (cₓₜ₉).
18. Explain why the pressure Pᵢ in this experiment is higher than in the previous experiment.
19. Explain why the flux in this experiment is lower than in experiment 2.

**Conclusions**
What are your conclusions based on the experimental results and discussion?