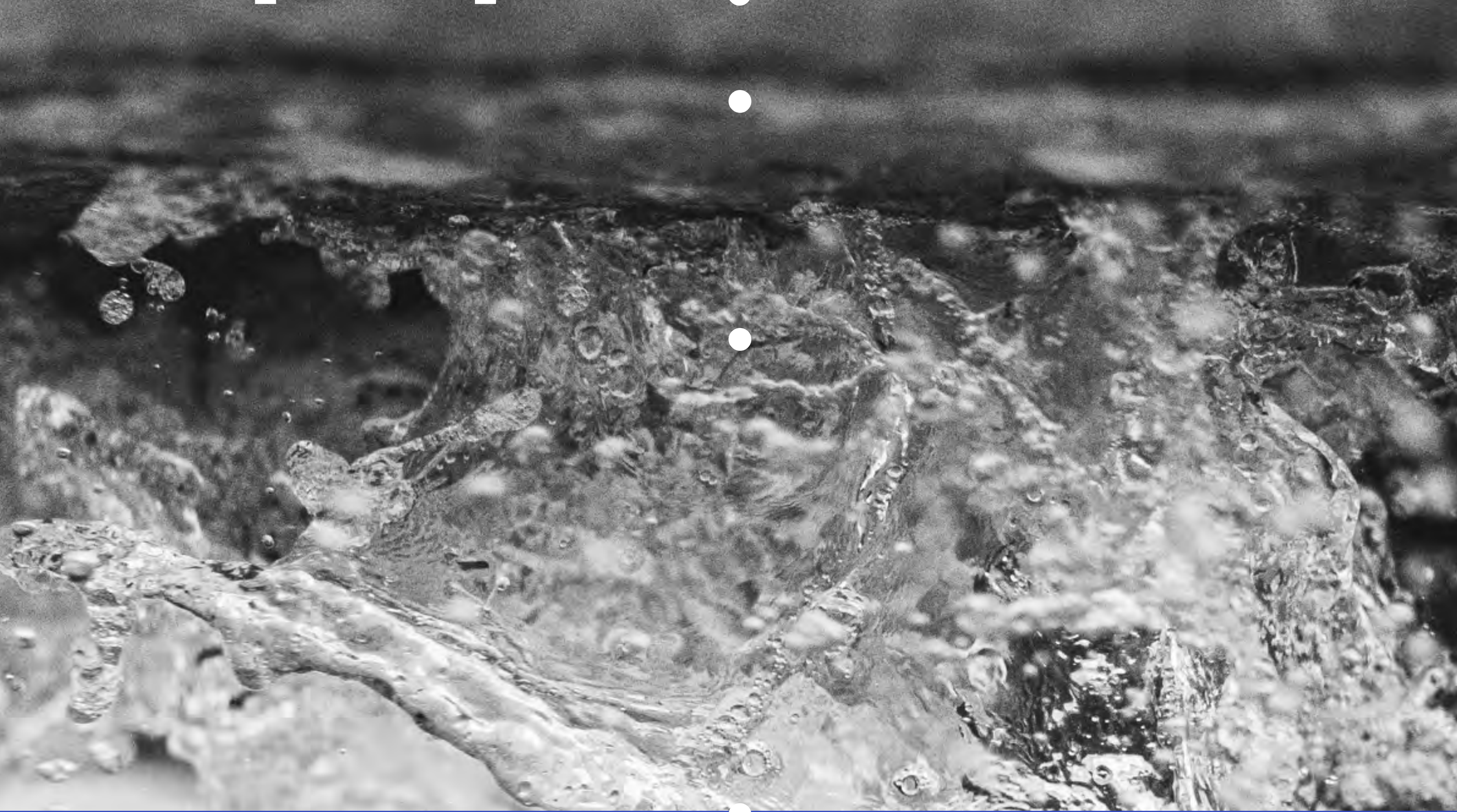
Aquaporin Inside®



Hollow Fiber Forward Osmosis

User manual

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Introduction

1.1 Purpose

This user manual contains general recommendations for installation, operation, maintenance and troubleshooting to sustain the performance of your Aquaporin Inside® Hollow Fiber Forward Osmosis (HFFO®) membrane module. The manual covers the modules HFFO®2 and HFFO®14. As the specifications and operational ranges vary between the HFFO®2 and HFFO®14 modules, refer to the datasheet for your module when studying the contents and guidelines in this manual.

1.2 Application and principles of forward osmosis

Forward osmosis (FO) is a membrane technique in which a semipermeable membrane is used to separate water from dissolved solutes. An osmotic pressure gradient across the membrane delivers the driving force to facilitate water transport from the feed solution (low osmotic pressure) to the draw solution (high osmotic pressure). Feed and draw solutions flow on the lumen and shell side of the membrane, respectively, without additional hydraulic pressure. As a result, the FO process allows the simultaneous concentration of the feed solution and dilution of the draw solution.

The dewatering potential of a feed solution in FO depends directly on the feed and draw solutions' quality and composition. Generally, the feed and draw solution's osmotic pressures (π), total dissolved solids (TDS), chemical oxygen demand (COD), total suspended solids (TSS), oil and grease (O&G) and detailed chemical composition are needed to evaluate the feasibility of FO technology.

1.3 Aquaporin Inside® architecture

The FO membrane is based on Aquaporin Inside® architecture. The semipermeable polymeric membrane consists of an active layer of Aquaporin Inside® plus a support layer. The essential building block of Aquaporin Inside® technology is the aquaporin water channel protein. Aquaporins are crucial for life in all organisms, from bacteria and plants to humans. They facilitate rapid, highly selective water transport across the cell membrane, allowing the cell to regulate its volume and internal osmotic pressure according to hydrostatic and/or osmotic pressure differences. The special architecture of the aquaporin channel allows only water molecules to pass; all other compounds are rejected.

1.4 Basic FO performance parameters

Membrane performance can be evaluated using the main parameters presented in Table 1:
Water flux (J_W, Equation 1), reverse solute flux (RSF, J_S, Equation 2) and specific reverse solute flux (SRSF, Equation 3). Water recovery (Equation 4) is an important FO process parameter. The driving force for the process is osmotic pressure (Equation 5).

Table 1: Equations describing membrane performance for feed batch and single-pass processes. V is feed batch volume [L]; Q is feed flow rate [L/h]; c is the draw solute concentration in the feed [g/L]; A is membrane area [m^2]; t is the duration of a batch concentration process [h]; δ is dead volume on the feed side of the system [L]; i is the dimensionless van't Hoff factor; C is the molar concentration of the solute; R is the ideal gas constant; T is the temperature [K].

	Batch mode on feed side	Single-pass mode on feed side	
Water flux, J_W	$J_W = \frac{V_{initial} - V_{final}}{A \times t}$	$J_W = \frac{Q_{F,inlet} - Q_{F,outlet}}{A}$	Eqn 1
Reverse solute flux, J_S	$J_S = \frac{c_{final} V_{final} - c_{initial} V_{initial}}{A \times t}$	$J_S = \frac{c_{F,outlet} Q_{F,outlet} - c_{F,inlet} Q_{F,inlet}}{A}$	Eqn 2
Specific reverse solute flux, SRSF	$SRSF = \frac{J_S}{J_W}$		Eqn 3
Water recovery, R	$R = 100 \times \left(1 - \frac{V_{F,final} + \delta}{V_{F,initial} + \delta}\right)$	$R = 100 \times \left(1 - \frac{Q_{F,outlet}}{Q_{F,inlet}}\right)$	Eqn 4
Osmotic pressure, π	$\pi = i \times C \times R \times T$		Eqn 5
Forward rejection, $Rej_{compound}$	$Rej_{compound} = 1 - \frac{c_{D,outlet} Q_{D,outlet} - c_{D,inlet} Q_{D,inlet}}{\frac{c_{F,outlet} + c_{F,inlet}}{2} \times (Q_{D,outlet} - Q_{D,inlet})}$		Eqn 6*
	$Rej_{compound} = 1 - \frac{c_{D,final} V_{D,final} - c_{D,initial} V_{D,initial}}{\frac{c_{F,final} + c_{F,initial}}{2} \times (V_{D,final} - V_{D,initial})}$		Eqn 7*

* Replace Equation 6 by Equation 7 to determine the forward rejection if the draw side runs in batch mode.

2

Packing, storage and transportation

2.1 Packing

The HFFO® modules are packaged safely to minimize risk of damage during transportation. It is recommended to save and reuse the original packaging if the modules are to be transported at a later stage.

2.2 Storage

2.2.1 Unused modules

Store unused modules in the original packaging, in cool and dry place out of direct sunlight or light.

2.2.2 Used modules

When out of operation for longer than one day, used modules should be unloaded from the skid or test setup and stored. Optimal storage conditions are 4 °C and out of direct sunlight or light to limit biological growth. If it is not possible to unload the modules from the skid or test setup, we recommend flushing the modules thoroughly with reverse osmosis (RO) permeate or deionized water (DI) water on both the lumen and shell side.

To prevent biological growth during system shutdown for longer than one week at ambient conditions or module storage for more than two weeks in cold storage (4 °C), the clean module can be filled on both lumen and shell side in a membrane preservative solution. It is recommended to use a 1 % Sodium metabisulfite (SMBS) solution up to 6 months. The pH of the SMBS solution must be monitored to avoid pH < 2. The membrane module must be cleaned to minimize fouling residues before preservation. Before taking stored modules back into operation, remember to thoroughly discharge excess preservatives by rinsing with water.

Always keep the module moist after any activity involving wetting, clean water performance testing and/or use in the FO application process (Section 5.2). Before storage, always clean a used module as described in Section 7 .

2.3 Transportation

Unused HFFO® modules should be shipped dry. They can tolerate freezing temperatures.

Used HFFO® modules should be filled with DI water on both the lumen and shell side before shipping. Freezing temperatures should be avoided during shipping.

Refer to Section 9.3 if the used HFFO® module is intended for membrane autopsy.

3

Installation & process configuration

3.1 Technical specifications

Please refer to the datasheet for your HFFO® module for technical specifications, characteristics, drawings and dimensions.

3.2 Module orientation

It is recommended to install and operate the modules in a vertical orientation, as rinsing and cleaning operations are more efficient when modules are placed vertically due to better air removal. If conditions do not allow for vertical orientation, the modules can be operated horizontally. In this case, precautions must be taken to prevent air from being trapped in the modules. Please refer to the datasheet for your HFFO® module for more information on the module's dimensions and connections for the feed and draw solution.

3.3 Process connections

The modules are designed to operate in 'FO mode', i.e. when the feed solution is facing the membrane's active layer.

To operate HFFO®2 in FO mode, the feed solution is connected to the module's central connection (lumen), while the draw solution is connected to the module's peripheric connection (shell).

To operate HFFO®14 in FO mode, the draw solution enters the module from the central connection, while the feed solution is connected through the eccentric thread (next to the central connection). Refer to the datasheet for a detailed description of the recommended feed and draw connecters for HFFO®14.

The connectors allow the operator to disconnect the module easily and limit any stress between the peripheric piping and module housing. The adapted O-ring can be adjusted to ensure a tight fit. We recommend using a hose rather than a pipe to connect to the module in order to allow flexibility and avoid stress points in the membrane inlets and outlets. The hose should be tightened with hose clips. The module connection threads should be wrapped with Teflon® tape or similar.

It is recommended to use plastic fittings to minimize stress and reduce the risk of damage to the fitting/ connection threading. Metal fittings are generally rough on the plastic threading when inserted and removed. Mixing of plastic and steel is not ideal because the materials behave differently when exposed to temperature, pressure and chemistry.

3.3.1 Spare parts

If spare parts, such as caps and O-rings, are needed for an HFFO®2 module, please contact sales@aquaporin.com. No spare parts are needed for HFFO®14.

3.4 Flow direction of feed and draw solution

The feed and draw solutions can be run in both co-current (parallel) or counter-current (opposite) flow across the FO membrane. Figure 1 shows the osmotic pressure difference (Δπ) in counter-current and co-current flow.

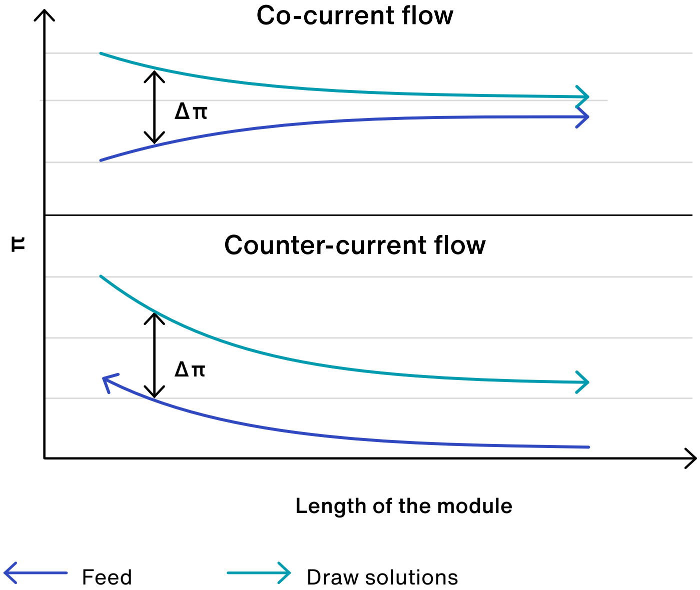


Figure 1: Theoretical osmotic pressure variations along the length of the module for feed and draw solutions run in co-current flow (top) and counter-current flow (bottom).

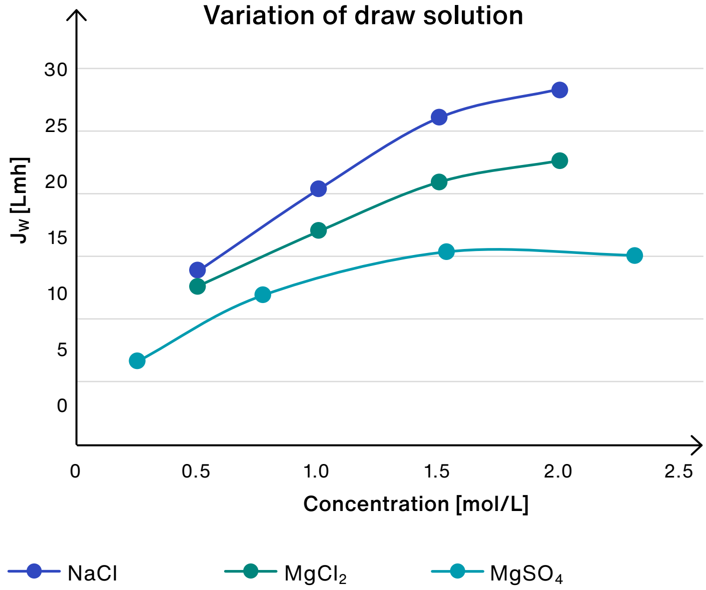


Figure 2: Water flux versus draw solute type and concentration for an HFFO®2 module when operating in standard FO conditions with DI water as a feed solution.

Counter-current flow is recommended in most operating situations, as it helps maintain a constant osmotic pressure difference between the feed and draw solutions within one membrane module. Counter-current flow has a number of other advantages:

- The driving force can be kept on a medium level over the full length of the module.
- The resulting local water flux (J_W) is more homogeneous over the length of the module.
- The arithmetic mean of the driving force over the full length of the module is higher compared to co-current flow, leading to better overall module performance.

Co-current flow is recommended if there is a risk of local negative transmembrane pressure (TMP) occurring inside the HFFO® module when operating in counter-current flow.

When operating a counter-current flow in a vertically orientated module with the feed flowing from bottom-up on lumen side and the draw flowing top-down on shell side, precautions must be taken to prevent air from being trapped in the module's shell side.

3.5 Draw solute type and concentration

The driving force for water transport in FO is based on the difference in osmotic pressure across the active layer. Figure 2 shows the relative effect of the solute type and concentration on water flux. Equation 5 can be used to calculate the related osmotic pressure for each solute type and concentration.

The expected water flux using a draw solution with identical molar concentrations of NaCl, $MgCl_2$ and $MgSO_4$ is higher for NaCl than $MgCl_2$ and is higher for $MgCl_2$ than $MgSO_4$. It is recommended to choose the minimum molar concentration of the draw solution necessary to reach the desired feed recovery and sustainable FO water fluxes.

3.6 FO process configuration

3.6.1 Batch and single-pass processes

Both the feed and draw solution can be operated in batch mode or in continuous (single-pass) mode (Figure 3).

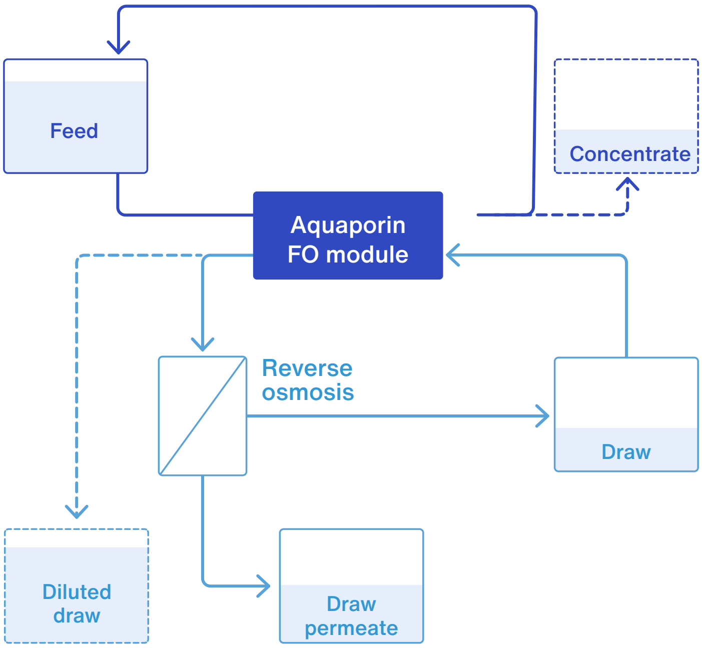


Figure 3: Schematic of single-pass operation (dashed line) and batch operation (solid line) for feed and draw solutions. RO draw regeneration is used as an example for batch operation of the draw solution.

Single-pass mode is used for most continuous processes. However, operating the feed side in batch mode and the draw side in single-pass mode – or vice versa – may be preferred in some cases.

Running the feed solution in batch mode enables higher concentration factors with less membrane area. A batch process is terminated when the required concentration factor or water recovery is reached or when the osmotic pressure of the feed solution equals the osmotic pressure of the draw solution.

Running the draw solution in single-pass mode ensures a constant driving force from the draw side. This operation mode is applicable when large volumes of draw solution (e.g. sea water) are available or only short tests are conducted. Alternatively, a draw regeneration step (for example, reverse osmosis in the draw loop) can be included to dewater the draw solution and keep the draw osmotic pressure constant in the FO process.

Refer to Table 2 for more information on how to choose the appropriate operation mode for your setup.

Table 2: Selection criteria for process configuration in single-pass or batch mode.

Criteria for process configuration	Single pass (feed and draw in single pass)	Feed in batch mode and draw in single pass	Batch (feed and draw in batch mode)
Feed and concentrate flow rates exceed practical tank sizes	✓		
Small feed flow rates and high concentration factors are desired		✓	✓
Discontinuous feed flow	✓ (if equalization tank is placed before FO)	✓	✓
Fluctuating feed composition	✓ (if equalization tank is placed before FO)	✓	✓
Maximizing driving force	✓	✓	
Large draw volume available	✓	✓	
Low membrane area requirements		✓	✓
Processing time limitations	✓		

3.6.2 Flow diagram and equipment for bench-scale and pilot-scale setups

HFFO®2 is suitable for lab/bench-scale installations. HFFO®2 and HFFO®14 are suitable for pilot-scale installations. HFFO®14 is suitable for full-scale operations (Table 3).

Table 3: Size of installations suitable for the respective HFFO® modules.

	HFFO®2	HFFO®14
Laboratory/bench scale	✓	
Pilot scale	✓	✓
Full scale		✓

The diagram in Figure 4 shows a general process flow, including the minimum required instrumentation and equipment to ensure successful operation of both bench-scale and pilot-scale setups. The diagram is applicable for HFFO®2 and HFFO®14 modules, although the flow conditions and piping size will vary depending on the size of the installed module.

Pilot systems are typically used for evaluating processes before scaling up to full-scale systems. The intended feed stream is concentrated in a continuous process to validate bench-scale results and enable a scale-up to industrial scale systems. It is possible to run pilot-scale FO processes in both single-pass and batch mode.

In the pilot setup, the pipe dimensions and material selection for the parts depend on the type of feed and draw solution, as well as the setup dimensions. A detailed parts list for a bench-scale and pilot-setup is presented in Table 4 and Table 5, respectively.

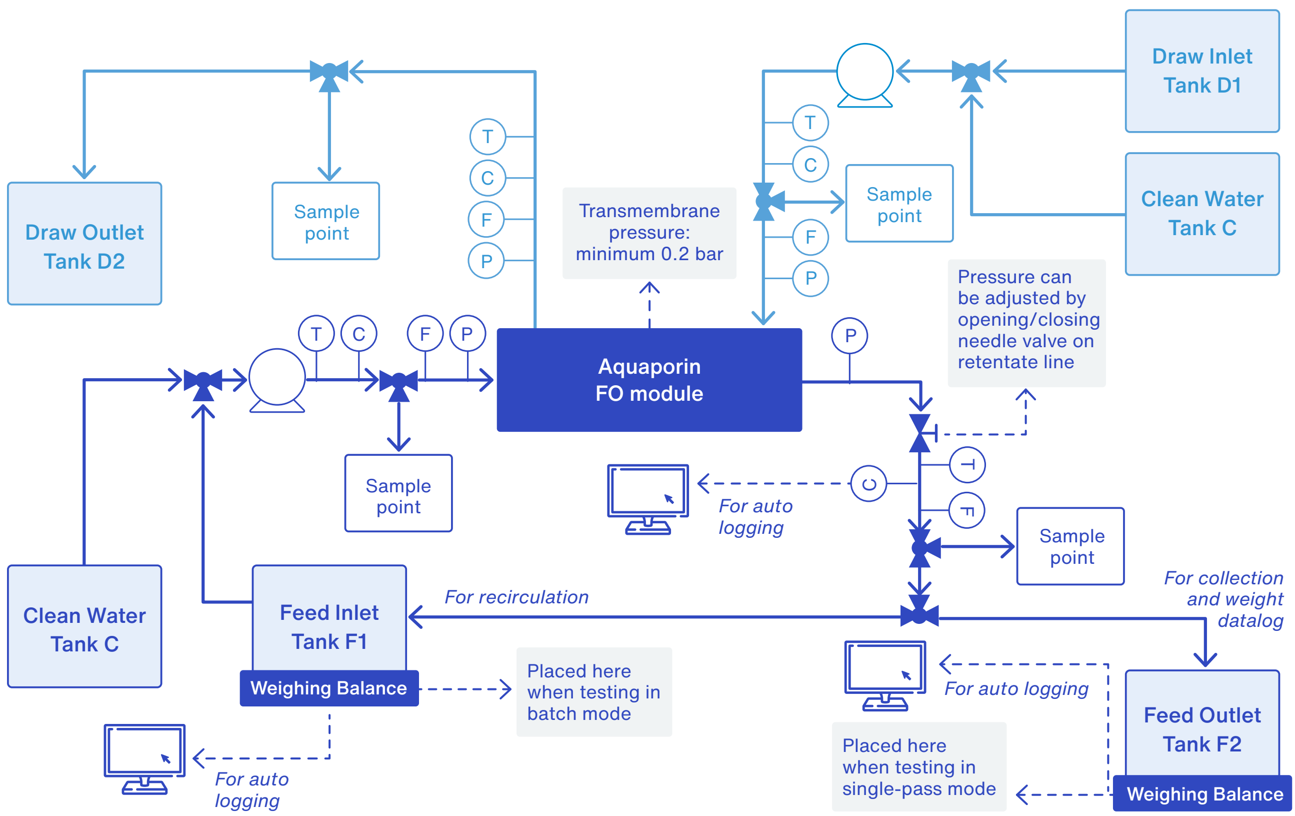


Figure 4: Instrumentation guideline for FO bench/pilot-scale setup for concentration tests for feed in batch and single pass and draw solution in single pass.

Table 4: Minimum required parts for a bench-scale setup for one module of HFFO®2.

Item	Quantity	Remarks
Conductivity meter*	1	Feed outlet (with auto logging)
Conductivity probe holder	1	For conductivity meter at feed outlet
Weighing balance	1	With auto-logging, maximum capacity 65 kg
Flowmeter	3	Analogue, 5 LPM max
Pump	2	Gear pump (min. 10 L/h; max. 180 L/h)
Pressure gauge	4	Analogue, 5 bar max.
Regulating valve	1	Feed outlet, TMP adjustment
Sampling valve	4	Feed inlet, feed outlet, draw inlet, draw outlet
Cartridge pre-filter	1	Feed inlet (max. 50 µm)
Laptop	1	For data recording
Collecting tank, 50 L	2	Feed inlet, feed outlet
Collecting tank, 100 L	1	Draw inlet
Collecting tank, 200 L	1	Draw outlet
Collecting bottle, 5 L	2	Feed and draw batch (for material compatibility testing)
Clean water tank, 50 L	1	For rinsing and cleaning purpose
CIP bottle, 5 L	1	For chemical cleaning purpose
Off-line pH meter	1	For sample analysis and CIP chemical preparation
Retort stands or clamps	1	For vertical placement of module
Fittings/tubing	n/a	
Alu-profile frame	1	~ 1,700 mm x 500 mm x 1,800 mm (LxWxH) for mounting instruments

*Automatic logging of temperature is recommended.

Table 5: Minimum required parts for a pilot-scale setup with one HFFO®14 module.

Item	Quantity	Remarks
Conductivity meter*	2	Feed outlet, draw inlet
Conductivity probe holder	2	For conductivity meters at feed outlet, draw outlet
Flowmeter	3	Feed inlet, draw inlet, feed outlet Digital, measuring range depending on pump dimensioning
Pump	2	Self-priming, - 300 L/h feed (down to 100 L/h to mitigate pressure drops) and 100 L/h draw*
Pressure transmitter	4	Digital, 5 bar max. Measuring at feed inlet/outlet, draw inlet/ outlet
Regulating valve	2	Feed outlet, draw outlet for TMP adjustment
Sampling valve	4	Feed inlet, feed outlet, draw inlet, draw outlet
Cartridge pre-filter	2	Feed (max. 50 µm)
PLC	1	For system control (HMI recommended)
Venting valve	2	Optional (to ensure that air is not trapped in the system)
Collecting tank, 200 L	2	Feed inlet, feed outlet
Collecting tank, 200 L	2	Draw inlet, draw outlet
Clean water tank, 50 L	1	For rinsing and cleaning purpose
CIP tank, 50 L	1	For chemical cleaning purpose
Off-line pH meter	1	For sample analysis and CIP chemical preparation
Fittings/tubing	n/a	For alimentary application
Pilot stand	1	Dimensions depend on parts' dimensions

**The max. pump flow capacity depends on number of HFFO® modules and array design. Feed composition and viscosity may require specific pumps and flow rates. Pump flow rates should also be able to cover wetting and clean water performance conditions, please refer to section 5.2.1 and 5.2.3, respectively.

For the described pilot-scale FO setup, a continuous supply of concentrated draw solution is required. This can be achieved by continuously supplying a freshly prepared draw solution. However, the large draw volume flows required by pilot units do not allow for long operation, as it is impractical to maintain large flows of freshly prepared draw solution. Therefore, installing a draw recovery unit is recommended. Additionally, DI or RO water will be needed for flushing, cleaning, disinfection and sanitation operations.

Aquaporin can support you with specific recommendations for the type of draw recovery unit suitable for your application. Please contact your Aquaporin sales representative or sales@aquaporin.com.

4

Understanding process parameters and feed quality

Before starting the FO operation (Section 5), it is important to understand the effect of feed water quality and different process parameters on FO performance. *It is critical for membrane performance that operation is maintained within the physical and chemical ranges specified in the datasheet for your HFFO® module.*

4.1 Process parameters

Varying process and operational parameters will increase or reduce key FO performance indicators. Table 6 shows a summary of the effects of parameter variation on the key indicators of water flux, RSF and SRSF. Each of the parameters is explained in greater detail in the subsequent sections.

A comprehensive description of the roles of process parameters during operation of Aquaporin Inside® HFFO® modules is published in the article: "Role of Operating Conditions in a Pilot Scale Investigation of Hollow Fiber Forward Osmosis Membrane Modules. Victoria Sanahuja-Embuena et al, Membranes (Basel). 2019 Jun 3; 9(6)."

4.1.1 Operating mode

The HFFO® modules are specifically designed for inside-out filtration (i.e. the feed solution is introduced on the inner (lumen) side of the hollow fibers and the draw solution is introduced on the outer (shell) side. This is known as FO mode. In FO mode, the feed solution flows on the membrane's active layer, which enables operation with maximal water flux.

The operating mode can be changed to PRO mode by switching which sides of the membrane the feed and draw solutions are introduced on. In this case, the draw solution will flow on the membrane's active layer. PRO mode is rarely recommended. Contact your Aquaporin Sales representative or sales@aquaporin.com for more information if considering operating in PRO mode.

Table 6: Influence of process parameters on Jw, RSF and SRSF (↑ increase, ↓ decrease, = no influence). The table does not consider synergy effects caused by modifying two or more parameters simultaneously.

Parameter	J_W	RSF	SRSF
1. Operating mode			
FO mode	↓	↓	=
PRO mode	↑	↑	=
2. Flow direction			
Co-current	↓	↓	=
Counter-current	↑	↑	=
3. Feed flow rate			
Increase	= / ↑	=	=/ ↓
Reduction	= / ↓	=	=/ ↑
4. Feed concentration			
Increase	↓	= / ↓	= / ↑
Reduction	↑	= / ↑	= / ↓
5. Draw flow rate			
Increase	↑	↑	=
Reduction	↓	↓	=
6. Draw concentration			
Increase	↑	↑	=
Reduction	↓	↓	=
7. Temperature			
Increase	↑	↑	=/↑
Reduction	↓	↓	=/↓
8. TMP			
Increase	↑	= / ↓	= / ↓
Reduction	↓	= / ↑	= / ↑

4.1.2 Flow direction

The suggested flow direction for HFFO® modules is counter-current for most applications. Counter-current flow is obtained by pumping the feed solution into the HFFO® module in the opposite direction of the draw solution flow. As described in Section 3.4, counter-current operation allows for maximum driving force across the entire length of the module and hence maximal water flux.

4.1.3 Feed flow rate and concentration

Generally, an increase in feed flow increases water flux due to a reduced effect of dilutive internal concentration polarization in the membrane support layer in the absence of fouling/scaling. The feed flow rate determines the hydraulic retention time of the feed solution inside the fiber lumen. At a given TDS in the draw solution, the lower the TDS concentration (hence osmotic pressure) in the feed solution, the higher the water flux (due to the higher osmotic pressure difference between the feed and draw solution).

4.1.4 Draw flow rate and concentration

A higher draw flow rate and concentration increase water flux. Increasing the draw flow rate reduces the retention time, minimizing the dilutive internal concentration polarization and thus the dilution of the draw solution along the module. The draw flow rate determines the hydraulic retention time in the module and therefore the amount of dilution along the module. An increase in draw concentration will result in a higher osmotic pressure difference between the feed and draw solution, increasing water flux.

4.1.5 Temperature

Temperature directly influences osmotic pressure (Equation 5) as well as the feed and draw solutions viscosity and diffusion coefficients. This means that both the water flux and RSF increase with an increase in temperature. However, SRSF should remain unaffected.

4.1.6 Transmembrane pressure (TMP)

Transmembrane pressure (TMP) is the average hydraulic pressure difference between the feed solution and the draw solution (Equation 8):

$$TMP = \frac{p_{Feed,in} + p_{Feed,out}}{2} - \frac{p_{Draw,in} + p_{Draw,out}}{2}$$

Equation 8

In general, it is highly recommended to operate with positive TMPs in order to ensure stable membrane performance. It has been observed that negative TMPs provoke an increase in Js/Jw and thus a loss in membrane performance.

4.2 Feed water quality

The exact nature, chemical composition and particle content of the water fed into the FO process will affect membrane performance and lifetime. While Aquaporin Inside® HFFO® membranes have high tolerances, certain chemicals may damage the membrane or result in fouling/scaling.

4.2.1 Chlorine

Under certain conditions, the presence of free chlorine and other oxidizing agents will cause premature membrane failure. It is recommended to remove residual free chlorine by pretreating with activated carbon or dosing with sodium bisulfite prior to membrane exposure. Refer to the datasheet for your HFFO® module for the maximum concentration of free chlorine.

4.2.2 Total suspended solids (TSS)

The presence of suspended solids in the feed stream will cause clogging of inlets and choking of fibers, resulting in an increased pressure drop, reduced water flux and potential fiber damage. It is recommended to reduce the absolute particle size to below 50 µm and concentration to below 30 ppm of TSS. This can be done by appropriate pretreatment such as bag filtration, media filtration, microfiltration or ultrafiltration. In all cases, it is recommended to use a < 50 µm cartridge filter at the feed inlet to protect the membrane.

4.2.3 Oil and grease (O&G)

The presence of oil and grease (O&G) could provoke severe fouling on the membrane fibers. It is recommended to reduce the concentration of O&G to < 20 ppm. Pretreatment strategies for O&G removal include air dissolved flotation (DAF), coagulation/flocculation, microfiltration and ultrafiltration.

4.2.4 Scaling and fouling

If the feed solution contains salts, the concentration process can cause local oversaturation of the solution, leading to scaling – especially in solutions containing salts with low water solubility (e.g. SiO_2, $CaCO_3$ and $BaSO_4$). Scaling can often be reduced or prevented by adding acids (which decrease oversaturation and solubilize certain precipitates) or anti-scaling agents (which decrease the precipitation level) to the feed solution.

Membrane fouling occurs in the presence of organic foulants which can also interact with inorganic compounds. Fouling requires regular membrane cleaning.

Heavy formation of fouling or scaling layers on the membrane surface decreases the membrane's performance and lifetime. Pretreatment of the feed solution, frequent membrane cleaning and the addition of anti-scaling agents or acids can help to optimize the process and prolong membrane lifetime.

5

Operating the module

The guidelines in this section refer to operation of a single HFFO® module. Similar recommendations apply for operating arrays consisting of multiple modules. Contact your Aquaporin sales representative or sales@aquaporin.com for questions regarding your specific array design.

5.1 Operating ranges

The physical and chemical operating ranges are different for HFFO®2 and HFFO®14. Refer to the datasheet for the specifications and operational limits for your specific HFFO® module. *To avoid damaging the module, it is critical that operation is maintained within the correct physical and chemical ranges.*

5.2 Start-up

5.2.1 First use: Wetting of the module

Wetting the HFFO® module is necessary prior to first use. Rinse the module with DI water for 30 minutes on both the feed and draw sides. Use low pump flow rates (Table 7) and ensure no overpressure in the feed or draw inlets.

Table 7: Minimum wetting flow rates.

Initial wetting flow rate, L/h	HFFO®2	HFFO®14
Lumen side	20	100
Shell side	10	50

5.2.2 Avoid osmotic drying

When starting any FO operation of HFFO® modules, DI or RO permeate water should flow on both the lumen and shell side before the feed solution is introduced to the lumen side.

Always introduce the feed solution prior to the draw solution to avoid osmotic drying of the membrane. The draw solution should only be introduced once a constant feed outlet flow has been reached (i.e. when the lumen is filled with liquid).

Adjust the inlet flow rate to ensure that both the feed and draw outlet flow rates never drop below the minimum outlet flow rates (Table 8).

Table 8: Minimum outlet flow rates.

Minimum outlet flow rate, L/h	HFFO®2	HFFO®14
Lumen side	10	50
Shell side	10	50

5.2.3 Clean water performance

For bench-scale applications, measuring clean water FO performance on a pristine and wetted HFFO® module is recommended. This enables you to establish a performance baseline. In order to achieve the performance stated on the datasheet for the given HFFO® module, the exact test conditions described in Table 9 must be used.

Table 9: Standard conditions for testing clean water performance of HFFO®2 and HFFO®14 in single-pass operation with a counter-current flow direction.

Product ID	Feed solution	Draw solution	TMP	Temperature
HFFO®2	DI water, 60 L/h	0.5 M NaCl, 25 L/h	0.2 bar	25 °C
HFFO®14	DI water, 400 L/h	0.5 M NaCl, 200 L/h	0.2 bar	25 °C

The test should be conducted in single-pass operation with counter-current feed and draw flow directions (Figure 5). After the instrument readings have stabilized, collect stable data for at least 30 minutes and evaluate the average water flux, RSF and SRSF. If the data do not correspond to the performance specified on the datasheet, verify that the test conditions are in accordance with Table 9.

When operating a used HFFO® module, measure clean water FO performance on the cleaned and flushed HFFO® module to verify FO performance integrity before starting the FO process. The performance should stabilize after a few uses. If performance has not stabilized after a few uses, your process or cleaning conditions may not be fully optimized.

5.2.4 FO process start-up

After establishing clean water performance (Section 5.2.3), the actual FO application with your specific feed water can be started. The feed inlet particle size should not exceed 50 μm. If the feed contains particles exceeding this size, install a 50 μm cartridge filter.

The initial FO process performance of used HFFO® modules may differ depending on storage conditions.

Only adjust to the desired TMP once flow and pressure in the module are stable (section 5.3.2). The FO process will operate in steady state as soon as the water flux and TMP are stable in single-pass configuration. The stabilization time depends on the specific application.

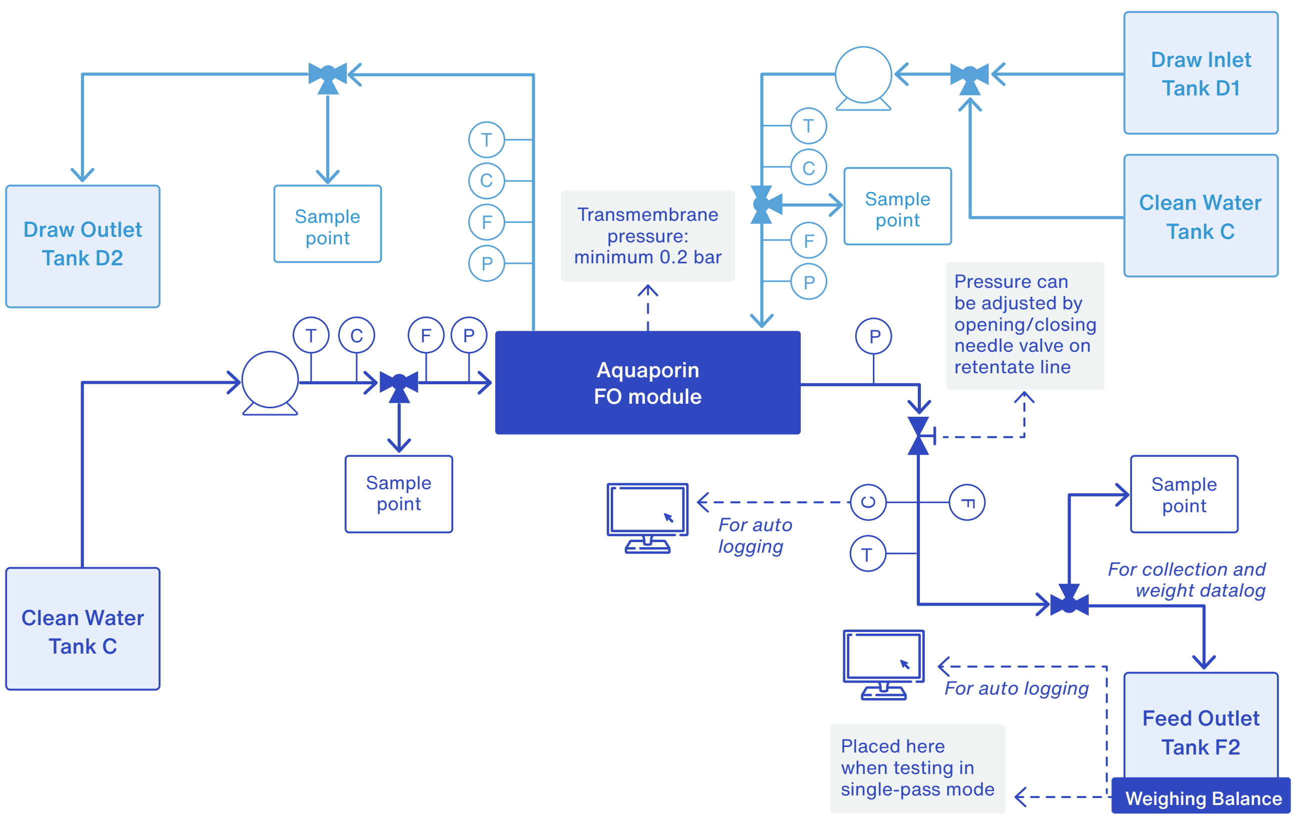


Figure 5: Instrumentation guideline for FO bench/pilot-scale setup for single-pass clean water performance tests.

5.3 General operation

5.3.1 Avoid dry operation

Do not allow the module feed outlet to run dry during any FO operation. Doing so may damage the membrane. The following precautions can be taken to prevent the module from running dry:

- Increase feed flow rate
- Decrease draw flow rate
- Decrease draw solution osmotic pressure

5.3.2 TMP adjustment

TMP is adjusted by closing or opening the feed outlet valve. This causes the feed line pressure to increase, which will lower the feed flow rate. The adjustment of the TMP should be slow. The feed outlet flow from the module must not be interrupted, as this may cause the module to run dry. Do not exceed the maximum allowed feed pressure for your HFFO® module (refer to the relevant datasheet).

5.4 Shutdown

To shut down the FO process, replace all liquids in the HFFO® module with DI water by rinsing the modules as follows in the list below; do not stop the feed and draw pumps during the FO process as it may cause over-recovery in fibers.

1. Reduce the draw solution flow rate to the minimum values (refer to Table 7).
2. Flush the feed line (lumen side) with DI or RO water by gradually increasing the existing flow rate to the recommended values for the lumen side (Table 10). A higher feed inlet rinsing flow may be required to stop the feed outlet flow from dropping below the minimum value (Table 8). If necessary, rinse the lumen side at a higher inlet flow rate; however, without exceeding the maximum flow rate given in Table 9.
3. After 15 minutes, or when the feed outlet conductivity and flow rate stabilize, flush the draw line (shell side) with DI or RO water at the minimum flow rate (refer to Table 7).
4. Increase the shell side flow rate gradually to the recommended values specified in Table 10. Always ensure a positive outlet flow on both the feed and draw side and ensure a positive TMP in the module. Proper rinsing is indicated by the equal conductivity and flow rates of the incoming and outgoing streams.
5. Shut down the shell side pump. Flush the lumen side for an additional 25 minutes with the shell outlet valves open to allow any permeating water to leave the module freely.
6. Shut down the lumen side pump.

Rinsing with clean water will not remove extensive membrane fouling or scaling. However, membrane lifetime can be prolonged by rinsing with clean water after each application run.

Table 10: Recommended (and max) RO/DI water rinsing flow rates.

Recommended (and max) RO/DI water rinsing flow rate, L/h	HFFO®2	HFFO®14
Lumen side	60 (max 150)	400 (max 900)
Shell side	25 (max 70)	100 (max 400)

6

Monitoring & performance evaluation

6.1 Monitoring

6.1.1 Data measurement

Table 11 lists the process parameters that should be measured during operation in order to evaluate the results obtained in pilot-scale installations.

Table 11: Process parameters to be measured during operation.

Process parameters	Where to measure	Purpose of measurement
Volume	Feed batch	Water flux (batch feed mode)
Flow rate	Feed inlet/outlet, draw inlet/outlet	Water flux (single pass feed mode)
Conductivity	Feed inlet/outlet*	RSF, draw concentration, osmotic pressure (indirect measurement)
Pressure	Feed inlet/outlet, draw inlet/outlet	TMP, feed line pressure drops, draw line pressure drops
Temperature*	Feed outlet, draw outlet	Temperature limit and fluctuation check
pH	Feed inlet, draw inlet	pH range within HFFO® product specifications and fluctuation check
Contaminant concentration	Feed inlet/outlet, draw inlet/outlet	Forward rejection

*Automatic logging of temperature during conductivity readings is recommended.

All data measurements should ideally be automatically and digitally logged for easy calculation and tracing. Aquaporin can provide support material for data logging to help you monitor your FO process. Please contact your Aquaporin sales representative or sales@aquaporin.com.

6.1.2 Performance parameters

Using the measurements outlined in Table 11, important online performance parameters can be calculated based on the equations shown in Section 1.4. These performance parameters and the necessary measurements are listed in Table 12. Calculation of these parameters should account for system dead volume where necessary. Other key performance parameters require offline analysis of the feed and draw compositions in order to be calculated.

Table 12: FO performance parameters.

Performance parameters using online measurements	Performance parameters using offline measurements
Water flux	Forward rejection
RSF	
Feed differential pressure	
TMP	

6.1.3 Sampling and analysis of feed/draw composition

Sampling of the feed and draw solutions is required during the process for offline analysis of the feed/draw composition. Please follow the below advice for good sampling practice.

- Only sample after stabilization of the FO process.
- The feed outlet sample should be taken before the feed inlet sample to minimize any feed flow disturbances.
- To minimize osmotic drying and sudden changes in the feed pressure and TMP during sampling in

the feed line, choose a sample valve with a small diameter and sample slowly.

- In batch operation, calculation of recovery should account for batch volume changes because of volume removal during sampling.
- Samples should be stored at 4°C until analyzed.

6.2 Evaluating performance

6.2.1 Water flux

Monitoring the water flux is important to ensure that the FO process is stable and operating satisfactorily. Rapid decreases in water flux from an otherwise steady flux evolution can be an indication of unwanted events impairing membrane performance.

Figure 6 shows a typical feed batch concentration process of secondary municipal effluent using a constant draw solution concentration of 1 M NaCl. The water flux decreases over time due to the steadily increasing osmotic pressure of the feed solution as well as formation of fouling and scaling layers. Severe fouling or scaling may occur at the end of a batch concentration process, due to both high external concentration polarization on the feed solution side and accumulation of foulants over the entire processing time.

Figure 7 shows a continuous concentration process of secondary municipal effluent using a constant draw solution concentration of 1 M NaCl. The decrease in water flux is expected, mostly due to fouling and/or scaling as the feed runs in single-pass mode and maintains a constant driving force. Initially the water flux decreases gradually, but at the point indicated by the grey box, the decrease in water flux accelerates. This acceleration indicates that the critical flux of the membrane has been reached.

In both Figure 6 and Figure 7, the dotted line projects how water flux will decline in the absence of fouling/scaling. In order to run the concentration process efficiently and to prolong membrane lifetime, it is recommended to clean the membrane before abrupt changes to water flux occur.

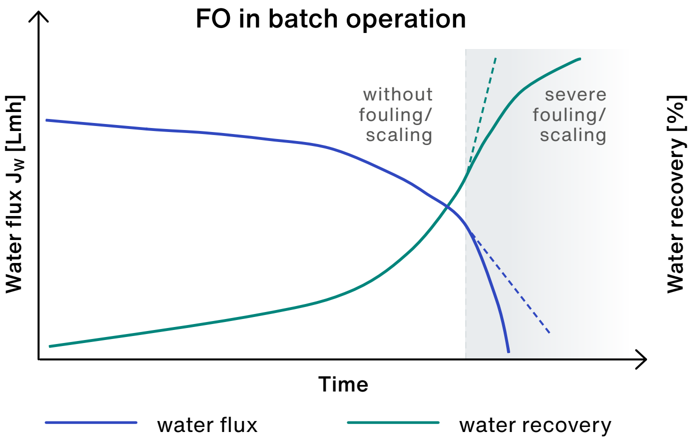


Figure 6: Water flux and water recovery as a function of time with (solid line) and without (dotted line) fouling/scaling during a batch concentration process with a constant draw solution concentration.

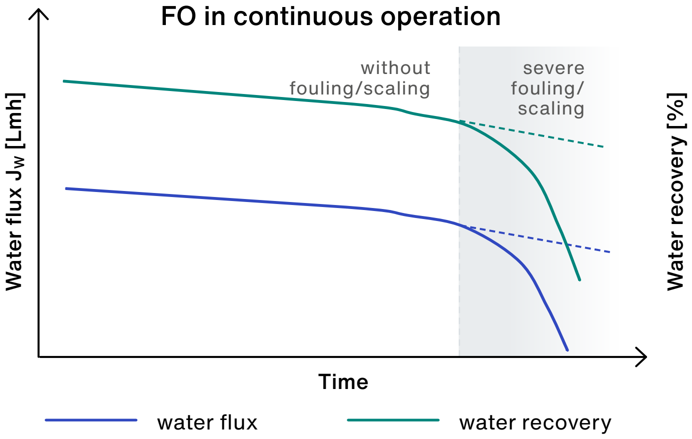


Figure 7: Water flux and water recovery as a function of time with (solid line) and without (dotted line) fouling/scaling during a continuous concentration process with a constant draw solution concentration.

6.2.2 Pressure drop

Feed pressure drop data can be used to evaluate process performance. An increase in pressure drop typically indicates that the membrane needs cleaning.

6.2.3 General evaluation advice

Always carefully monitor process parameters and look for changes in normal parameter trends. Sudden spikes, deviations or rate changes to process parameters compared to their normal behavior in steady state conditions suggest abnormal evolution of the process characteristics, which may be rooted in faulty equipment, membrane deterioration or erroneous operation.

7

Cleaning and sanitation

7.1 General cleaning

Fouling and scaling occur on membrane surfaces due to the presence of salts, such as calcium, and organic and biological matter. Fouling and scaling include any type of material deposition on the membrane surface. It can affect membrane performance, typically by lowering permeate flow and increasing solute passage. Cleaning can often restore initial membrane performance.

The following precautions should be taken to avoid any degradation of the membrane caused by cleaning:

1. Remove any process solution from the system and flush the module with DI water, as explained in Section 5.2.1.
2. Use DI or RO water rather than tap water for flushing, cleaning, disinfection and sanitation when possible. Failure to do so can compromise membrane performance and shorten its lifetime.
3. Allow permeation by opening the shell side to atmospheric pressure while applying a cleaning solution on the lumen side of the module only.
4. Ensure the cleaning solution is mixed thoroughly before circulating in the module.
5. Always stabilize the temperature before dosing pH to chemical cleaning solutions, as a change in temperature induces a change in pH. Continuously monitor pH and temperature during cleaning to ensure that the temperature and pH do not exceed the limits for the specific HFFO® module. *These limits are stated in the relevant module datasheet.*
6. Never clean the membrane with cleaning agents that contain free chlorine. To protect the HFFO® module's selective membrane layer from oxidation, upstream de-chlorination of the membrane by sodium bisulfite dosing or activated carbon is required.
7. Cleaning chemicals may be present on the lumen and shell side after cleaning. Ensure any residuals are properly flushed from the module and system.

7.2 Cleaning-in-place (CIP)

Cleaning-in-place (CIP) is conducted to remove reversible organic fouling and scaling. Reversible organic fouling can be removed using alkaline cleaning solutions containing alkaline agents, such as NaOH, KOH, etc. Reversible inorganic fouling or scaling can be removed using acidic cleaning solutions containing acidic agents, such as citric acid, HNO_3, HCl, etc. CIP process ranges are different for each HFFO® module. Keep within the operational limits stated in the datasheet for your HFFO® module to avoid irreversibly damaging the module.

7.2.1 CIP of HFFO®2

The general procedure for chemical cleaning HFFO®2 modules consists of the following steps:

1. Recirculate alkaline cleaning solution at an elevated temperature for 20 minutes separately in the feed line and draw line. Separating the draw and feed lines during cleaning is necessary to avoid contamination.
2. Flush both the feed and draw line with ambient temperature DI water until neutral pH is achieved at the feed and draw outlet. Always start feed line flushing before simultaneous draw flushing to avoid drying the feed lumen.

3. Recirculate acidic cleaning solution at an elevated temperature for 20 minutes separately in the feed line and draw line. Separating the draw and feed line during cleaning is necessary to avoid contamination.
4. Flush both the feed and draw line with ambient temperature DI water until neutral pH is achieved at the feed and draw outlet. Always start feed line flushing before simultaneous draw flushing to avoid drying the feed lumen.

Refer to Table 7 and Table 10 for flow ranges suitable for rinsing or CIP. To identify the effect of each cleaning step on membrane performance, test and evaluate the FO clean water performance as described in Section 5.2.3.

7.2.2 CIP of HFFO®14

The general procedure for chemical cleaning of the HFFO®14 module consists of the following steps:

1. Remove any process solution from the system and flush the module with DI water as explained in Section 5.2.1.
2. Use DI or RO water rather than tap water for flushing, cleaning, disinfection and sanitation when possible. Failure to do so can compromise membrane performance and shorten its lifetime.
3. Allow permeation by opening the shell side to atmospheric pressure while applying a cleaning solution on the lumen side of the module only.
4. Ensure the cleaning solution is mixed thoroughly.
5. Introduce a continuous flow of DI water on the shell side with a minimum flow rate of 200 L/h.
6. Introduce the cleaning solution on the lumen side at a maximum flow rate of 50 L/h and maximum temperature of 40 °C.
7. Allow the CIP to run for 20 minutes while closely monitoring the pH level of the continuous flow of DI water to ensure the shell outlet stays within the maximum pH range (refer to the HFFO®14 datasheet).
8. Drain the cleaning solution from the system and flush the module with DI water as described in Section 5.2.1 to reach neutral pH.

During CIP, always closely follow the flow rates stated above. This is to ensure that the pH of the CIP solution stays within the operational limits in the datasheet.

7.3 Sanitation (HFFO®2)

Heat (Section 7.3.1) and hydrogen peroxide (Section 7.3.2) sanitation procedures can be applied to HFFO®2 modules in order to reduce the amount of bacteria. It is recommended to remove any type of deposit on the membrane by CIP before sanitation. Sanitation guidelines for HFFO®14 are currently being developed by Aquaporin.

7.3.1 Heat sanitation

The following steps can be followed to sanitize the module via recirculation of warm DI water:

1. Simultaneously heat and circulate warm DI water (≈85 °C) in the feed and draw line, keeping the two lines separate. The heating rate should not exceed 5 °C per minute.
2. Maintain the temperature for at least 30 minutes.
3. Decrease the temperature after sanitation at a maximum rate of 5 °C per minute.
4. Flush the system with DI water.

7.3.2 Hydrogen peroxide sanitation

Follow the steps below to sanitize the module using hydrogen peroxide:

1. Alkaline and acidic cleaning should be completed before hydrogen peroxide sanitation to remove organic deposits and iron deposits from the membrane surface.
2. Dilute the hydrogen peroxide stock solution to 0.2-0.25 % using DI water or RO permeate. The presence of iron or other transitional metal in tap water will degrade the polyamide layer of the FO membrane when hydrogen peroxide is present.
3. Simultaneously circulate the 0.2-0.25 % hydrogen peroxide solution for 20 minutes in the feed and draw line separately at less than 25 °C.
4. After cleaning, comprehensively flush the system with DI water or RO permeate to ensure any residual hydrogen peroxide is removed.

8

Disposal

Used HFFO® modules can be disposed of as municipal waste if the module contains no hazardous solution. Consider possible deposits of hazardous substances from the feed inlet on the membrane before disposal.

9

Trouble-shooting

The most common problems encountered in FO applications are the decline of water flux and RSF. If a change in membrane performance is detected, it is recommended that a clean water test is performed (Section 5.2.3) to determine whether the HFFO® module has been damaged during operation. To determine the significance of FO performance deterioration, the measured water flux and RSF should be compared to the initial FO performance values measured before the FO application test. If these data are not available, measured water flux and RSF can be compared to datasheet FO performance.

A slow decrease in water flux and/or an increase in RSF can indicate sustained fouling formation. In this case, clean the module as described in Section 7.

A dramatic change in performance might indicate damage to the module. It is essential to take measures as early as possible to stop damage, as any delay can impede restoration of the module.

9.1 Diagnosis based on clean water performance

Perform a clean water performance test on the module (Section 5.2.3). If a significant change in performance is detected, check the following to verify the standard test setup:

1. Are all pressure, conductivity and flow meters and sensors properly calibrated? Accuracy of flow measurements can be verified via the mass balance equation:

   $$\text{Feed inlet} + \text{Draw inlet} = \text{Feed outlet} + \text{Draw outlet}$$

2. Has the system stabilized?

If the data are verified, check the following:

1. Check the feed quality in terms of particles, chlorine or other oxidizing chemicals.
2. Check if all chemical cleaning steps were performed within operational pH and T limits as stated on the datasheet for the relevant HFFO® module.

If all verifications are positive, the trends below can be interpreted/explained as outlined in the Sections 9.1.1-9.1.3.

9.1.1 Low water flux

A reduction of water flux is often caused by fouling/scaling. In addition, an increase and/or decrease in RSF might occur. In both cases, the module should be cleaned as described in Section 7.

9.1.2 High water flux

A sudden dramatic increase in water flux can indicate damaged fibers. Contact your Aquaporin Sales representative or sales@aquaporin.com for support.

9.1.3 High reverse solute flux (RSF)

A high RSF can be caused by fouling/scaling or fiber damage. In case of a simultaneous low water flux, chemical cleaning as per Section 7 is recommended.

9.2 Diagnosis based on FO process performance

9.2.1 High pressure drop

A high pressure drop on the feed inlet can indicate a blockage. If operating with HFFO®14, cleaning as described in Section 7 is recommended to restore membrane performance. If operating with HFFO®2, open both caps and inspect the feed inlet visually. If any clogging is visible, remove the layer and proceed with chemical cleaning. If the feed stream is particle loaded, install or replace the cartridge filter.

9.2.2 Leaking modules

Contact your Aquaporin Sales representative or sales@aquaporin.com for support if leakage of a HFFO®14 module is observed. Do not attempt to open the module.

If leakage from HFFO®2 is observed, perform the following steps to identify the location of the leak.

1. Piping connections: Ensure a proper seal between the connectors. Replace the connectors if damaged.
2. Caps:
   a. Ensure the caps are properly fastened.
   b. Remove both caps and check if the O-rings are in the right position. Close both caps and check if the module still leaks.
   c. Replace the O-rings.
3. Housing: Ensure the housing is not cracked. If the housing is cracked, the module must be replaced.

9.3 Membrane autopsy

If troubleshooting (Section 9) and cleaning (Section 7) cannot restore water flux or RSF, the membrane may be irreversibly damaged. Membrane autopsy performed by Aquaporin may reveal the root cause of module failure. Based on this, Aquaporin may recommend changes in your operation to prevent future module failures.

When returning a module for autopsy, the module should be emptied of liquids and the end caps should be closed. Each module can by identified by a unique lot/batch number on the module housing. When shipping several modules, please communicate these numbers to Aquaporin.

Contact your Aquaporin Sales representative or sales@aquaporin.com for more information.

10 Abbreviations and symbols

10.1 Abbreviations

DI	Deionized water
FS	Feed solution
DS	Draw solution
RSF	Reverse solute flux
SRSF	Specific reverse solute flux
TMP	Transmembrane pressure
CIP	Cleaning-in-place
TDS	Total dissolved solids

10.2 Symbols

J_W	Water flux
J_S	Reverse solute flux
J_S/J_W	Specific reverse solute flux
π	Osmotic pressure
$\Delta\pi$	Osmotic pressure difference

11 Notice and disclaimer

11.1 General recommendations & liability

Aquaporin scientists and engineers with in-depth knowledge of the membranes and their operation have prepared the recommendations in this document. However, any recommendations herein should be considered of a general nature and are for informative purposes only. It is the user's responsibility to ensure appropriate usage of this product. Aquaporin A/S assumes no obligation, liability or damages incurred for the misuse of the product or for the information provided in this document. This document does not express or imply any warranty as to the merchantability or fitness of the products. Nothing in this document is to be construed as recommending any practice or any product in violation of any patent, law or regulation.

11.2 Advisors

Aquaporin technicians present at a customer's site are solely to be considered as advisors, who are in no way responsible for the duties of the operations manager or responsible for operating the customer's facility. These responsibilities remain with the customer.

We wish to underline the importance of carefully reviewing the operating recommendations issued by Aquaporin before adopting them.

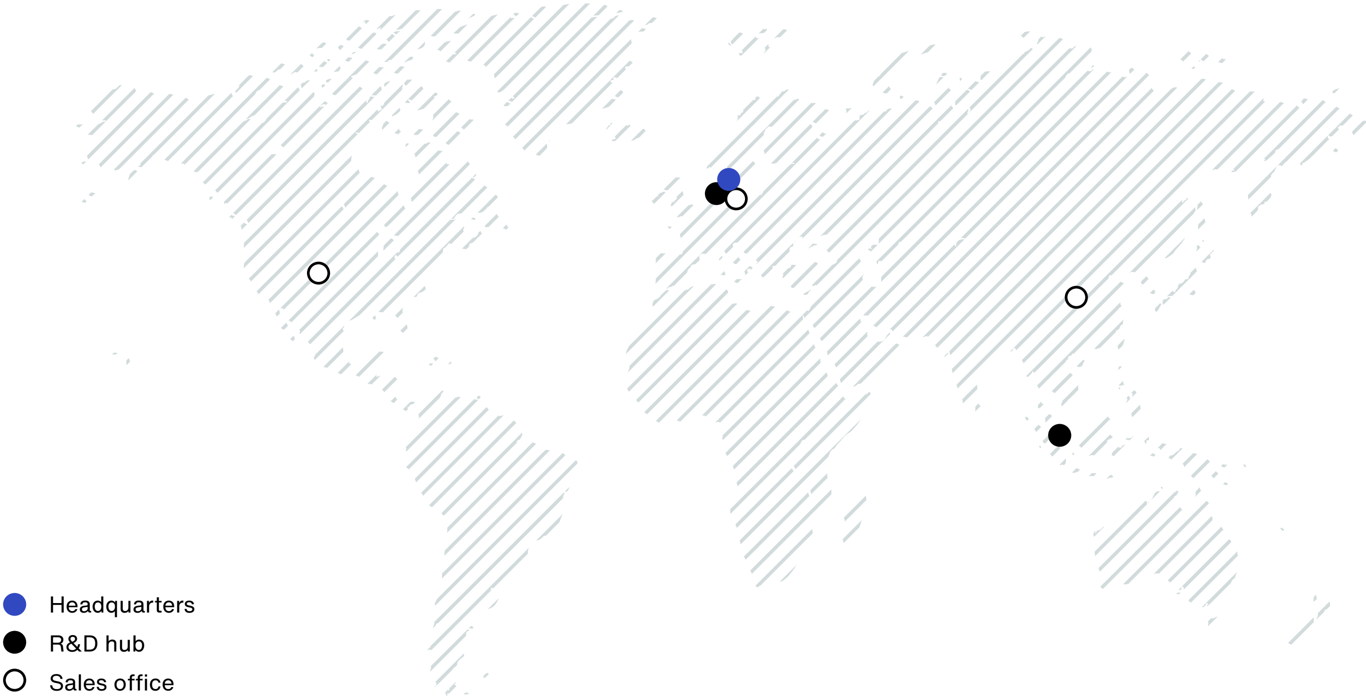
11.3 Unclear points

Unclear points, if any, should be discussed and clarified with Aquaporin before the start of operation.

Aquaporin at a glance

Aquaporin is a water technology company dedicated to natural water treatment with operations in Denmark (HQ), Singapore and the United States. At Aquaporin, we're working to preserve the Earth's most valuable resource – water – by combining advanced engineering, biotechnology and natural aquaporins – nature's own water purifiers – which we embed into water purification membranes. Our proprietary technology, Aquaporin Inside®, is based on Nobel Prize-winning research and used to clean and reuse water in industries, in our homes and even by NASA in space. We work with customers and partners around the globe to sustainably treat industrial wastewater, concentrate food & beverage products and enhance drinking water quality and accessibility.

Learn more at aquaporin.com.



Aquaporin A/S
Nymøllevej 78
2800 Kongens Lyngby
Denmark

Phone: +45 8230 3082
sales@aquaporin.com
aquaporin.com

Aquaporin Inside®