The non-biological on-site treatment system

Final report for MUDP project

Miljøteknologisk Udviklings- og Demonstrationsprogram

Projektet, som er beskrevet i denne rapport, er støttet af Miljøteknologisk Udviklings- og Demonstrationsprogram (MUDP) under Miljøministeriet, der støtter udvikling, test og demonstration af miljøteknologi.

MUDP investerer i udvikling af fremtidens miljøteknologi til gavn for klima og miljø i Danmark og globalt, samtidig med at dansk vækst og beskæftigelse styrkes. Programmet understøtter dels den bredere miljødagsorden, herunder rent vand, ren luft og sikker kemi, men understøtter også regeringens målsætninger inden for klima, biodiversitet og cirkulær økonomi.

Det er MUDP's bestyrelse, som beslutter, hvilke projekter der skal modtage tilskud. Bestyrelsen betjenes af MUDP-sekretariatet i Miljøstyrelsen.

MUDP-sekretariatet i Miljøstyrelsen
Tolderlundsvej 5, 5000 Odense| Tlf. +45 72 54 40 00

Mail: ecoinnovation@mst.dk
Web: www.ecoinnovation.dk

Denne slutrapport er godkendt af MUDP, men det er alene rapportens forfatter/projektlederen, som er ansvarlige for indholdet. Rapporten må citeres med kildeangivelse.

Contents

1. Introduction

Climate changes with more heavy rain, urbanisation, increased centralization and increasing environmental legislation demanding less sewer overflows, are putting an ever-increasing stress on the sewer systems all over the world. Sewer systems being the bottleneck in wastewater handling are very expensive and troublesome to maintain and expand.

In addition, widespread sewer network and centralization in large treatment plants does not support a sustainable use of water. Fresh water resources are taken from a large geographical area, collected through the sewer network, and often discharged into a marine recipient. This leaves small water streams with less or no water flow which again results in less biodiversity. In addition, water scarcity is a widespread problem, and the non-potable re-use of treated wastewater is recognized as the way to save on drinking water resources and secure drinking water for all. Though, with a centralized infrastructure, the re-use of treated water will require a completely new distribution network to the consumers, which is also very expensive and troublesome.

At present the economic challenges by far supersedes the capabilities of the sector.

Therefore, municipalities and wastewater service companies all around the world are in need for better, cheaper solutions, which can be scaled much faster.

The non-biological on-site wastewater treatment system aims to provide such an alternative, with a new decentralized solution for individual homes.

The idea is illustrated in FIGURE 1. Wastewater is collected in a traditional septic tank. Over time clean water is taken out of the septic tank and rejected substances are up-concentrated in the septic tank. After some time, the septic tank is emptied, and the content sent for anaerobic digestion at a central wastewater treatment plant. Then the up-concentration in the septic tank starts all over again, so over time it works like a batch process.
A feed pump (a) will recirculate wastewater from the septic tank to the FO membrane and return concentrate to the septic tank. A salt solution in the draw circuit will draw clean water out of the wastewater through the FO membrane. This will dilute the salt concentration, so to maintain the salt solution concentration, a high-pressure pump (b) will feed the draw solution to the RO membrane. Clean water will leave the system as permeate from the RO membrane and the up-concentrated salt solution (brine) will recirculate back to the FO membrane. The pressure energy recovery device (c) will reduce the outlet pressure from the RO membrane and use the recovered energy to save energy on the high-pressure pump (b).
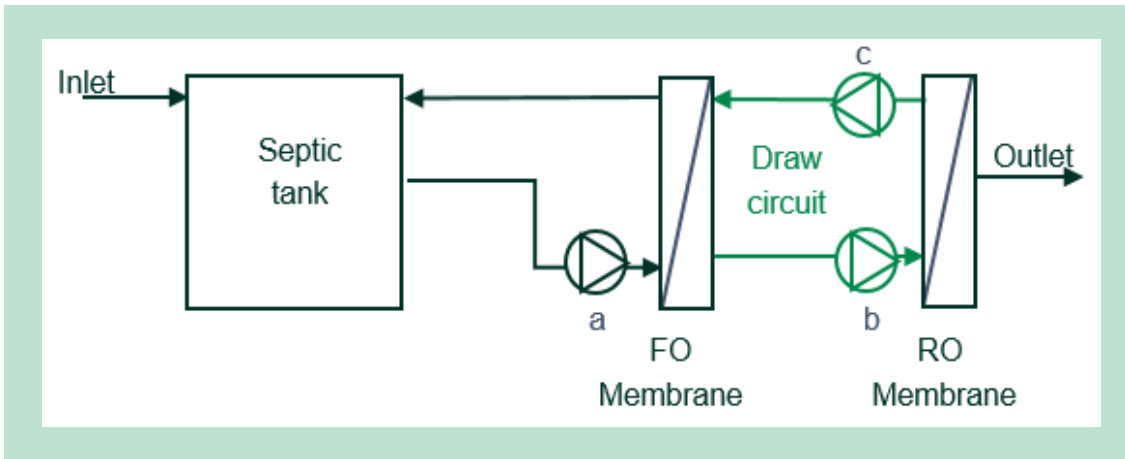
FIGURE 1. Plant diagram with main components.

This constitutes a completely new type of on-site treatment system, one that is physical-mechanical. This has the advantage of enabling cost-effective service operations of many systems and the recovery of more organic material for energy recovery via biogas production, as it does not rely on biological processes. It has advantages such as a higher treatment efficiency that allows treating wastewater to drinking water quality levels. This is vital as it enables installation in areas with high groundwater tables because it eliminates the risks from the outlet in relation to human health and groundwater protection, but also allows for water reuse in the household for non-potable purposes.

The project is led by Soholm Water Systems in collaboration with Aquaporin and DTU Sustain.

2. Executive summary

The idea of a non-biological on-site treatment system combining a septic tank directly with a FO-RO membrane filtration step to create a very high quality of water outlet is possible.
The project has shown that the FO and RO membranes provides a safe barrier against harmful bacteria and the outlet water quality is very high - at drinking water level. The water quality enables installation in areas with high groundwater tables and the water can be re-used for non-potable purposes such as toilet flushing, dishwasher, laundry machine and garden watering. The physical-mechanical working principle results in a much higher biogas potential in the retained sludge and a very low emission of greenhouse gasses. The water recovery from the septic sludge has proven so high, that the frequency of emptying the septic tank can be reduced to once a year.
The process has endeavored strong patent possibilities, which are now under filing process.

The purpose of the project is to verify the idea of a non-biological on-site wastewater treatment system and gain the knowledge and the technology that will enable the production of a prototype and clarify any needs for further technology development before said prototype can be realized.

The project has been realized during a combination of lab tests and testing on a pilot plant fed with domestic-like wastewater from the Herlev Hospital in Denmark.

The project has demonstrated that the idea of combining a septic tank directly with a FO-RO membrane filtration step to create a very high quality of water is possible.

The process in-fact is a slow developing batch process more than a continuously running process. The batch process starts when the septic tank is emptied and runs until the septic tank is emptied again. During this time an up-concentration of solids and substances will take place in the septic tank. This will also change the loading concentrations on the FO-RO membrane system during this period.

Lab results has shown that an ultimately 99% up-concentration of domestic wastewater is possible using FO membrane technology. This corresponds to that retained substances from 274 m^3 of treated wastewater is concentrated in just 2.7 m^3 septic tank volume. This means that 1-year intervals between emptying the septic tank is possible, even if maximum loaded every day of the year.

The pilot plant tests have demonstrated that a septic tank can work really well in holding back hair, fibres, fats, oils as well as heavier-than-water particles and create a feed quality for the FO membrane with a low level of suspended solids and particle sizes below 0.3 mm.
Much of the test run period lasting close to 365 days has been carried out using the same .0.2 mm SEFAR filter at the outlet of the septic tank. Still, this has not, according to more particle

size testing, had any impact of relevance on the feed quality going to the FO membrane. Though, practical experiences and lab tests have shown that larger particles may form after the septic tank and before the FO membrane over time and during stand-still. Therefore it is advisable that any such additional protective filter is placed closest possible to the FO membrane inlet.

Other filters were tested with nominal sizes from 0.1-0.15 mm, in different materials, non-woven as well as woven – all with poorer results. Especially the filter in polypropylene 0.1 mm blocked immediately upon contact with wastewater, which is assumed to be due to a mismatch in zeta potentials of the wastewater and the filter.

The 0.2 mm ID Aquaporin FO membrane type HFFO14 with 13.8 m^2 membrane area tested experienced clogging several times, mainly due to particle size exclusion at the inlet and is not considered appropriate for operation on septic tank effluent. Additional reduction of the larger particle sizes is needed and attachments of particles to the membrane surface will block the narrow hollow fibers very fast.

The 0.8 mm ID Aquaporin FO membrane prototype with 6.8 m^2 membrane area has been tested with better results. Visual inspections of the membrane inlet and outlet have not shown significant cake layer build-up. Only minor and easy-removable deposits have been seen.

During operation a head loss builds up inside the FO hollow fibers when wastewater is fed to the membrane. The head loss is caused by particles forming a cake layer on the membrane surface that narrows down the Inner Diameter of the Hollow Fibres. Electrostatic interactions between the membrane surface and wastewater particles, which again is highly influenced by salt concentrations in the wastewater, is also assumed to impact the membrane fouling/cake layer formation.
Tests have shown that this head loss/cake layer formation is rather independent on operating flux, when operated at low water fluxes around 2 LMH and low laminar feed flow between 100-400 L/hour.

Relaxation is a good strategy for controlling cake layer formations on the 0.8 mm Aquaporin membrane. Repeatedly, periods of standstill have resulted in a decline in the FO membrane head loss / reduction of cake layer. The longer the standstill period the bigger the decline. In addition to this the zeta potential of the membrane in relation to the wastewater needs to be addressed correctly, when designing the final system.

With the current 0.8 mm ID FO membrane a balance point seems to be present around 100 L/hour wastewater feed rate and 2 times 30 minutes relaxation per day.

The optimal flux will need longer time testing before this can be concluded finally, but operations around 2 LMH has so far been demonstrated. Lab tests indicate that a long-term sustainable flux will be lower than 4 LMH.

Biocide (Neuthox® 420 ppm pH 7.2) has been introduced to the FO membrane on a weekly basis (2 x 1 min) and the last three months (2 x 10 min) during testing to prevent biofilm growth. This has turned out to be much more than needed, so it is possible to optimize this further. Different types of biocides have been tested for chemical compatibility with the Aquaporin membrane and tests have shown more possible candidates, but also that the pH is very important and must be around 7 in order not to change membrane characteristics. Ozone, even at a small dose of 2 ppm, caused severe damage of the membrane and is therefore not a useful alternative.

Direct coupling of the FO and RO membrane with the same draw recirculation system has turned out well as it is able to balance itself, so RO flux equals FO flux after few hours of operation. This self-balancing is important as it makes it possible to make a simple system with low needs for controls and regulators.

Salt concentration in the draw, RO recovery rate and flux and RO operating pressure are all interconnected, so changes to one of these parameters will automatically change the others. Therefore, using a high-pressure pump on the RO inlet with a mechanically direct-coupled pressure energy recovery device on the RO outlet will be a way to lock the recovery rate and RO flux without the need for any further regulator or controls. When salt concentration goes up, power consumption of the pressure pump goes up, but system flows remain constant. Only thing left is the control of salt-addition to the system to maintain and increase the salt concentration needed over time. This shall be based on the level in the draw tanks.

Specific salt consumptions per final outlet produced were measured to 2.3-gram NaCl L^{-1} and 0.5-gram MgCl2 L^{-1} on one specific day. Especially the line operating on NaCl was suffering from issues on both the FO and the RO membrane during that day. Expectations for the final system is below 0.5-gram L^{-1} for both NaCl and $MgCl_2$.

The tests showed that H2S forms occasionally in the septic tank, and it affects the outlet quality with levels up to 0.6 mg S2- L^{-1}. A simple solution was found to this problem. By introducing a waterfall effect when the draw recirculation returns to the draw tank, -naturally aerated with atmospheric air - the problem was eliminated.

Tests have shown that an outlet water quality of very high quality and similar to drinking water level can be achieved with the system. An outlet water quality entirely free from phosphorous and low on organics and nitrogen. 108 pharmaceuticals, 16 PAHs, 7 heavy metals, 4 halogenated organics, 3 alkylphenols and 16 PFAAs have been tested and shows a general trend below limit of detection for almost all substances tested or below PNEC for those above.

The system membranes (FO and RO) provide a safe barrier against harmful bacteria and viruses in the wastewater due to size exclusion over both membranes. No E-coli or coliforms have been detected in the outlets.

Based on the test results and recommended system optimizations, evaluations of the outlet water quality of the final system have been made.

The expected outlet water quality is considered suitable for infiltration into the ground without risk of groundwater contamination and for direct discharge to receiving water recipients, where it can help reach targets for chemical and ecological state.

The expected water quality is considered suitable and safe for the intended non-potable purposes:
- Toilet flushing,
- Laundry and dishwasher machines,
- Garden watering, surface wash and car wash.

The outlet water will be aggressive/corrosive, due to the lack of calcium and too much carbonic acid and occasionally chloride content above drinking water limits. The water is only intended supplied via separate and new installations and therefore this corrosion risk must be mitigated by appropriate material selections. Today's machines for dishwashing and laundry washing are already designed for working with similar water qualities, so the corrosion risk here is assumed low.

In addition, re-using the water is expected to bring some extra benefits in daily use:
- Less detergents needed due to low level of carbonates.

- Lack of calcium will reduce bacteria smell issues in laundry washing machines.
- The aggressive water will remove calcium-based scaling when used for toilet flushing.

The biomethane potential of the concentrated sludge after 99% water recovery was measured to 648 ml CH4 gVS^{-1}, which is much higher than the typical levels around 200 ml CH4 gVS^{-1} reported for municipal secondary sludge. The tests show an overall very low biological activity level in the septic tank, both anaerobic and aerobic. This indicates a low degradation of the biomethane potential over time.
Almost no methane has been detected and a documented low level of ammonium transformation indicates a low level of nitrous oxide too – proving an overall very low emission of greenhouse gasses.
The biomethane potential tests show that some time to adapt to a higher salinity level in the anaerobic digester might be needed, before the full biomethane potential can be released.

The key components, FO membrane, RO membrane and high-pressure pump and pressure energy recovery device are all market available technologies. The technologies are present, but not available, so they are optimal for this specific system. The system is rather small in capacity; therefore, efficiency losses means relatively much and therefore key components must be optimized to reach the targets for performance, power consumption and cost.
The FO-membrane must be optimized in size and length/diameter ratios to fit in. Hollow fiber ID must be enlarged for robustness/reduction of clogging risk. Membrane surface might be optimized in terms of their zeta potential to reduce fouling and reverse salt flux to reduce salt losses.
The RO membrane must be optimized in size and length/diameter ratio to support 40-50% recovery rates.
The high-pressure pump and energy recovery device must be reduced in capacities to match the low flows. They must fulfil noise requirements and the suction side static pressure conditions in the final system design.

All these elements will have significant impact on the power consumption of the final system. Therefore, power consumption has not been measured as part of this project, as this would be far from relevant anyways. Though, learnings pointing towards general low flows and high recovery rates on the RO part in the final system, supports expectations for an attractive power consumption on the final system.

3. Project purpose

The purpose of the project is to verify the idea of a non-biological on-site wastewater treatment system and gain the knowledge and the technology that will enable the production of a prototype and clarify any needs for further technology development before said prototype can be realized. For this, the following objectives are set out:

- Test and develop ideas for how the wastewater should be taken out from the settling tank for membrane filtration, so that the operation of the forward osmosis (FO) membrane is not blocked by hair, fibres, and larger particles.
- Test and develop ideas concerning the integration and operation of the feed side of the FO membrane. In order to find the optimal combination of membrane area, flux, and cleaning needs, the focus is on:
  - Membrane design: Interior diameter (ID) and membrane area.
  - Minimum crossflow necessary to maintain functionality without blockages, with stable performance and at full capacity.
  - Cleaning needs: Frequency and cleaning agents.
- Test and develop ideas related to the integration and operation of the draw solution and the reverse osmosis (RO) membrane in order to determine the conditions necessary to keep the plant running at full capacity between service intervals and with the desired cleaning efficiency.
- Test ideas regarding the recirculation of the draw solution between the FO and the RO membranes and the relevant pumps, with a focus on the lowest possible power consumption.
- Gain knowledge, through analyses and measurements during the operation of the test plant, about which parameters and measuring points are best suited to monitor the plant and verify its correct function.
- Identify the need for further technology development through the acquired knowledge and experience before a prototype and later a market-ready solution can be realized. This will create the basis for a precise budget and plan for the next phase.
- Define a goal for the requirements for the plant's treatment efficiency and water quality at the outlet and designate the measuring points that can be used to verify that the system is working properly. This will be done based on knowledge of the current legislation in Denmark and abroad.
- Assess the expectation of treating the concentrated substances in the settling tank and in the draw solution at large central treatment plants, and their potential therein in relation to resource recovery, for instance through biogas.

4. Target and test site

4.1 Basic target

Salt will be lost through the FO membrane and the RO membrane during operation/filtering and therefore must be added over time.

A target is that the system can operate with service intervals of 1 year, as this is required as a minimum by law for small residential Onsite Wastewater Treatment Systems, but also as this will be required for the system to be an economically viable solution.

Consequently, the system will be working in a kind of batch mode, as the septic tank concentration will keep increasing during the period between emptying.

An Onsite Wastewater Treatment System designed for 5 person equivalents in Denmark must have capacity to treat the loads as indicated in TABLE 1.

TABLE 1. Basic capacity requirements for a 5 PE Onsite Wastewater Treatment System in terms of flow, chemical oxygen demand (COD), biological oxygen demand (BOD5), nitrogen load (N load) and phosphorus load (P load).

Parameter	Value
Specific flow per PE (L PE^{-1} d^{-1})	150
Maximum daily flow (L d^{-1})	750
Maximum annual flow (m^3 d-1)	274
COD load (g COD PE-1 d-1)	120
BOD load (g BOD5 PE-1 d-1)	60
N load (g total N PE-1 d-1)	13
P load (g total P PE-1 d-1)	2.5

Given that most septic tanks for 5 PE have volumes up to around 2.7 m^3, this means that the system ultimately in worst case scenario should be able to reach the following targets:

$$\text{Water recovery rate} = \frac{V_w - V_s}{V_w} \cdot 100 = \frac{274 \text{ m}^3 - 2.7 \text{ m}^3}{274 \text{ m}^3} \cdot 100 = 99\%$$

where V_w is the target volume to be treated (TABLE 1) and V_s is the volume of the septic tank.

4.2 Test site

The Herlev Hospital wastewater treatment plant was selected as test site. The plant has been in operation since 2014 and receives all the wastewater from the hospital. Rain and surface water is separated and does not influence on wastewater characteristics. This means that the wastewater is not diluted in any way before entering the wastewater treatment plant. Therefore, the concentrations of COD, BOD, suspended solids (SS), N, and P are high and comparable to the outlet from a home. In addition, the wastewater contains high levels of pharmaceuticals, bacteria and some environmentally hazardous substances that enables verification of the treatment efficiency of the system. Finally, the wastewater is well known and has been characterised in previous projects (see TABLE 2).

TABLE 2. Average values for the characteristics of the influent wastewater to Herlev Hospital wastewater treatment plant [1].

Nutrients (mg L^{-1})	
COD	773
Total N	66
Total P	13.1
Pharmaceuticals (ng L^{-1})	
Azithromycin	893
Ciprofloxacin	13486
Clarithromycin	2650
Diclofenac	646
Erythromycin	1005
Ifosfamide	1987
Sulfamethoxazole	5336
Iomeprol	2890000
Paracetamol	352000
Bacteria	
Antibiotic resistant bacteria	High concentration (10^6 -10^7)
Norovirus	High concentration ($1.7 \cdot 10^5$)
Other hazardous substances (µg L^{-1})	
Lead	4.0
Cadmium	0.079
Chromium	2.1
Copper	110
Mercury	<0.05
Nickel	4.8
Zink	100
EDTA	<3000
LAS	1600
DEHP	23
Bisphenol A	3.1

5. Laboratory tests

Before completing the design of the test plant, several laboratory tests were conducted.

5.1   Pre-filter test

When starting the project, only the Aquaporin 0.2 mm internal diameter-based FO membranes were available for the project.

Assuming that some additional filtering would be needed on the septic tank effluent to avoid clogging of these small diameter FO membranes due to solids, hair, and fibres during operation on the test plant, we did several experiments in the lab.

Raw wastewater from the Herlev Hospital wastewater treatment plant was taken out and allowed to settle for 24 hours and was then manually filtered to obtain a water quality representative of that, which could be used for up-concentration by the FO membrane. Prefiltration was carried out manually at DTU using commercially available Fibertex membranes with pore sizes 0.03, 0.065, 0.07 and 0.1 mm, to assess which prefiltration is suited for this particular wastewater and to make the 0.2 mm internal diameter FO membrane work for the tests.

Pore size 0.03 mm was immediately discarded due to excessive clogging during the prefiltration step.

The pre-treated water was then filtered using a HFFO2 Aquaporin FO module. The test setup (FIGURE 2) consisted of a feed tank with prefiltered wastewater placed on a scale, an FO module, and a draw solution tank. Conductivity was measured both in the feed and in the draw solutions and was logged continuously. The weight data from the scale were used to calculate the water flux (Jw), the net volume of water that crosses the membrane per unit area and time, and the feed recovery, the percentage of the feed that is recovered as clean water.
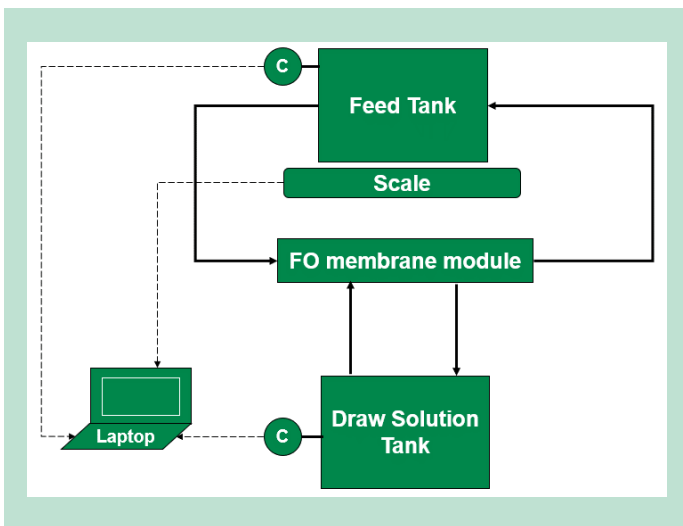
FIGURE 2. Test setup for the evaluation of prefilter pore size and FO membrane suitability.

Results of the pre-filter experiments:
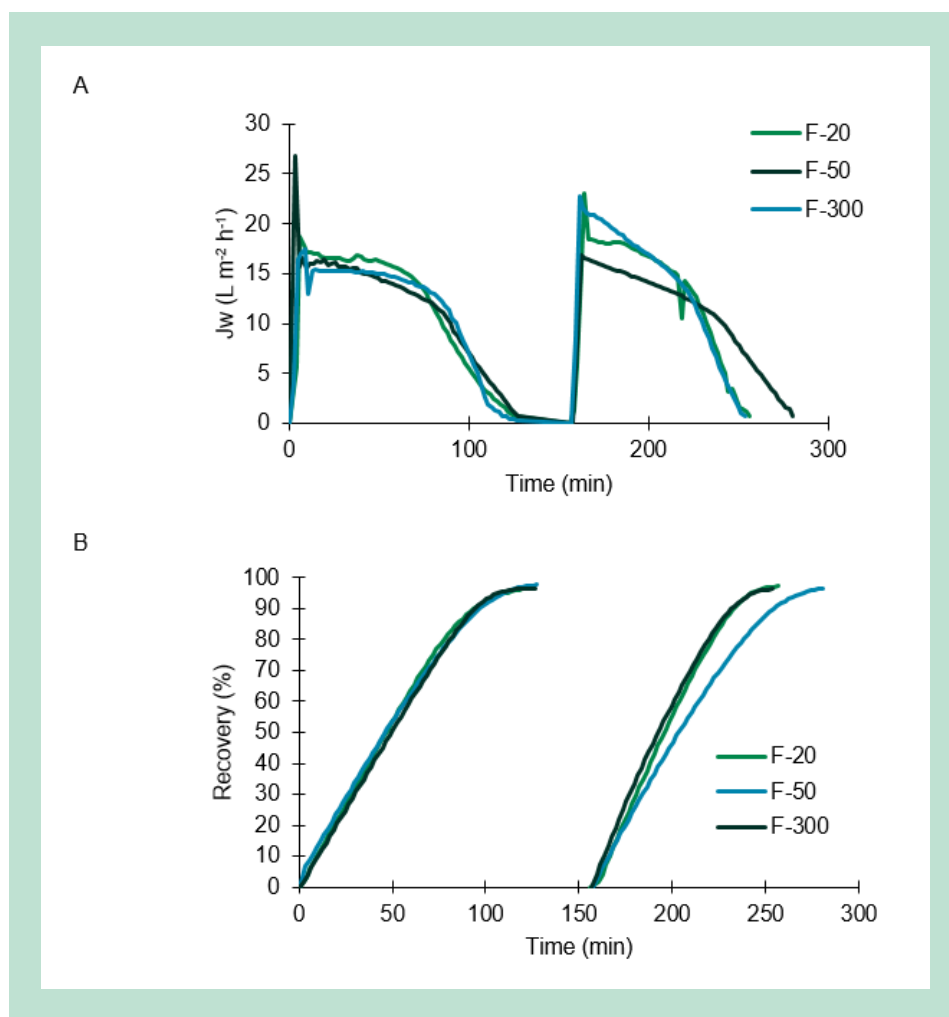
FIGURE 3. A) Water flux (Jw) over time, with 30-minute rinsing between 130 and 160 minutes, and quality control (QC) tests with deionised water at the start, at 160 minutes and at the end. B) Feed recovery (%) as a function of time. Commercial Fibertex membranes F-300, F-50, and F-20 with respective nominal pore sizes of 0.065, 0.07, 0.1 mm were used.

The conclusion of the experiments was that all three Fibertex filters generated an acceptable feed quality for the Aquaporin 0.2 mm internal diameter FO membrane when operated up to 98% recovery rates.

The water recovery curve slope starts to decrease after 85% recovery as the difference between the osmotic pressure of the draw solution used gets closer to that of the up-concentrated feed.

With similar results we opted for the F-20 with 0.1 mm nominal pore size as pre-filter1 going ahead, assuming that the largest pore size would work better in the test plant.

5.2   Maximum recovery test

Once it was established that 98% water recovery rate could be reached, a test was carried out to assess if the target recovery rate of 99 % was achievable.

Going from 98% to 99% recovery rate, may not sound like a lot, but for a system with a 2.7 m^3 septic tank, 98% corresponds to 135 m^3 of treated wastewater and 99% correspond to 274 m^3 of treated wastewater, double the volume, therefore relevant to investigate.

In addition, we wanted to obtain information about the characteristics of the feed concentrate when 99% water recovery had been reached. It was also important to observe if there was a significant increase in feed viscosity that could block the FO membrane before the target up-concentration was reached. Raw wastewater pre-treated with the 0.1 mm pore size Fibertex F-20 and the HFFO2 Aquaporin FO module were used for up-concentration.

The test setup can be seen in FIGURE 4. Weight of the feed buffer tank and feed conductivity before and after the FO module were logged continuously. The test was run in duplicate, and the feed concentrate was characterised in terms of total solids (TS), total suspended solids (TSS), volatile solids (VS) and volatile suspended solids (VSS) and osmolarity (mOsmol/kg).
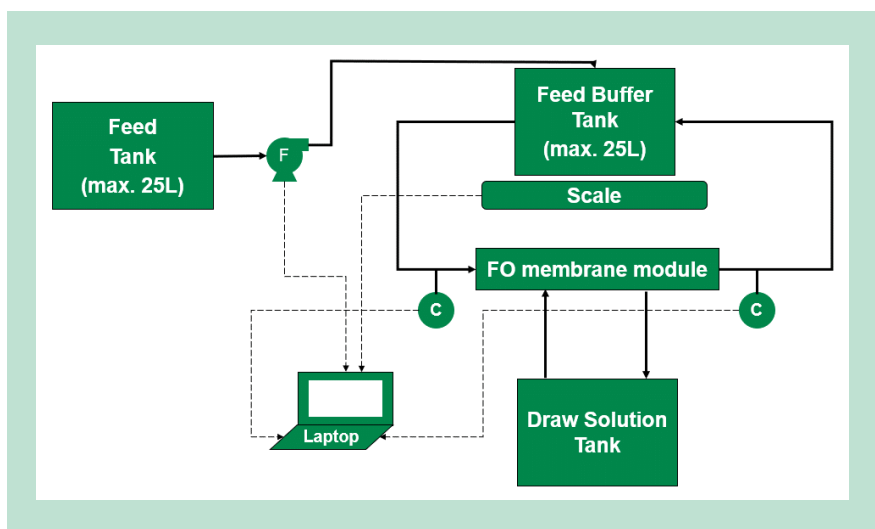
FIGURE 4. Test setup for the maximum recovery essay.

Each set started with app. 25 L of feed in the feed buffer tank. Starting from around 110 min (test run 1) and 500 min (test run 2) and the following 70 min – more feed was added with 0.346 L min^{-1}. In total 50080 ml were concentrated to 529 ml in test run 1 and 47794 ml were concentrated to 582 ml in test run 2. Corresponding to 98.9% and 98.8% water recovery ratio. 3x25 L of draw solution 1 M NaCl was used for test run 1 and 4x25 L of draw solution 1 M NaCl for test run 2. The corresponding developments in water recovery rates and draw inlet conductivities can be seen in FIGURE 5.
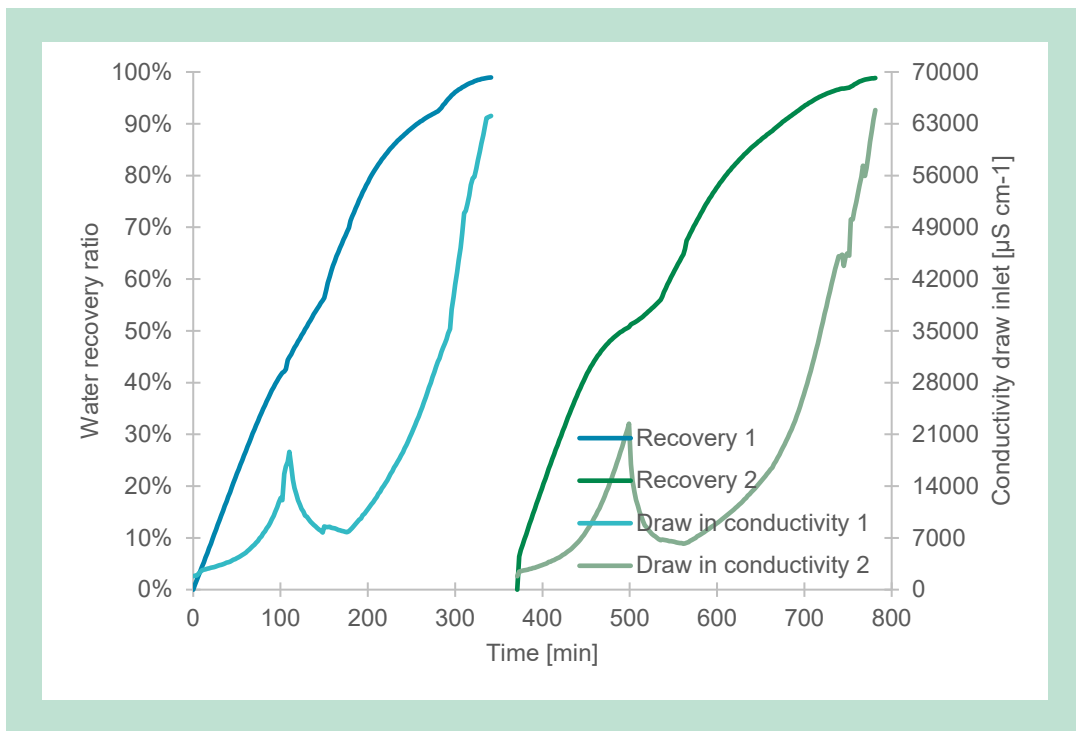
FIGURE 5. Maximum recovery test run 1 and 2.

TABLE 3. Physico-chemical characteristics of the concentrated feed after 99 % recovery had been reached.

Parameter	Start	End run 1	End run 2
TS (g L^{-1})	1.33	100.03	99.71
TSS (g L^{-1})	0.3	13.60	13.46
VS (g L^{-1})	-	41.27	40.99
VSS (g L^{-1})	-	6.37	6.18
Osmolarity (mOsmol kg^{-1})	34	1639	1653
Osmotic pressure at 20°C (bar)	0.8	39.9	40.2

The viscosity of the feed was not measured, but appearance of the viscosity of the concentrated feed was like water – no visual change was noticeable.

Based on the results, it was concluded possible to reach a maximum recovery rate of 99%.

The final osmotic pressure of the feed concentrate is around 40 bar. The draw solution will have to be higher in osmotic pressure to overcome the resistance in the FO membrane. This means that the transmembrane pressure required for the RO membrane will be higher than 41 bar (600 psi) which is the typical limit for Brackish Water type of RO membranes and therefore it can be concluded that Sea Water type of RO membranes will be needed (typical limit 69 bar (1000 psi) max operating pressure) for the system.

The parameters related to solids content are relevant for the subsequent use of the concentrate for valorisation through anaerobic digestion, and the osmotic pressure is a key factor for the design of the draw solution.

5.3   Salt selection and flux tests

The salt used in the draw solution should be an affordable, soluble inorganic compound with a low reverse salt flux on the FO membrane. Sodium chloride (NaCl), magnesium chloride ($MgCl_2$) and sodium bicarbonate ($NaHCO_3$) were evaluated.

Each salt will require different concentrations (and osmotic pressures) to achieve the same flux on the FO membrane. The required osmotic pressure differs from compound to compound due to their chemical nature, like charge and atom size. A higher osmotic pressure results in higher operating pressure for the RO membrane, which again means a higher power consumption for the system.

A low reverse salt flux is important for two reasons. The total amount of salt that must be added to the system increases at higher reverse salt fluxes. This makes system service more troublesome and costly. In addition, this salt loss to the feed increases the osmotic pressure of the feed, which again increases the osmotic pressure of the draw solution, which ultimately leads to higher power consumption for the system.

The performance of the salts was evaluated in terms of water flux (*Jw*), reverse salt flux (*Js*) and ammonia rejection.

Raw wastewater pre-treated with the 0.1 mm Fibertex F-20 and the HFFO2 Aquaporin FO module was used for the test. All tests were done with 25 L feed. Draw solutions were applied in single pass mode. The test setup can be seen in FIGURE 1.

Conductivity measurements were used as a proxy for osmotic pressure of the draw solutions. The osmotic pressure of a liquid is given as:

$$\Pi \text{ [bar]} = \text{Osmolarity } [\frac{\text{Osmol}}{\text{kg}}] \cdot 8310 \cdot (273 + T \text{ [°C]} \cdot 0.00001$$

where Π is the osmotic pressure and *T* is the temperature.

The correlation between osmolarity and salt concentration is as follows (example using NaCl): The molecular weight of NaCl is 58.44 g mol^{-1} and two atoms per NaCl molecule results in 2 osmol per mol NaCl.
1% NaCl wt wt^{-1} solution equals 10,000 mg L^{-1} NaCl when density is approximated 1 kg L^{-1}.

Osmolarity = 10,000 mg L^{-1} / 1 kg L^{-1}/ 1,000 mg g^{-1} / 58.44 g mol^{-1} x 2 osmol mol^{-1} = 0.342 osmol kg^{-1}

The correlation between salt concentration and conductivity were approximated, using data from CRC handbook and equations fitted using Matlab. See FIGURE 6.
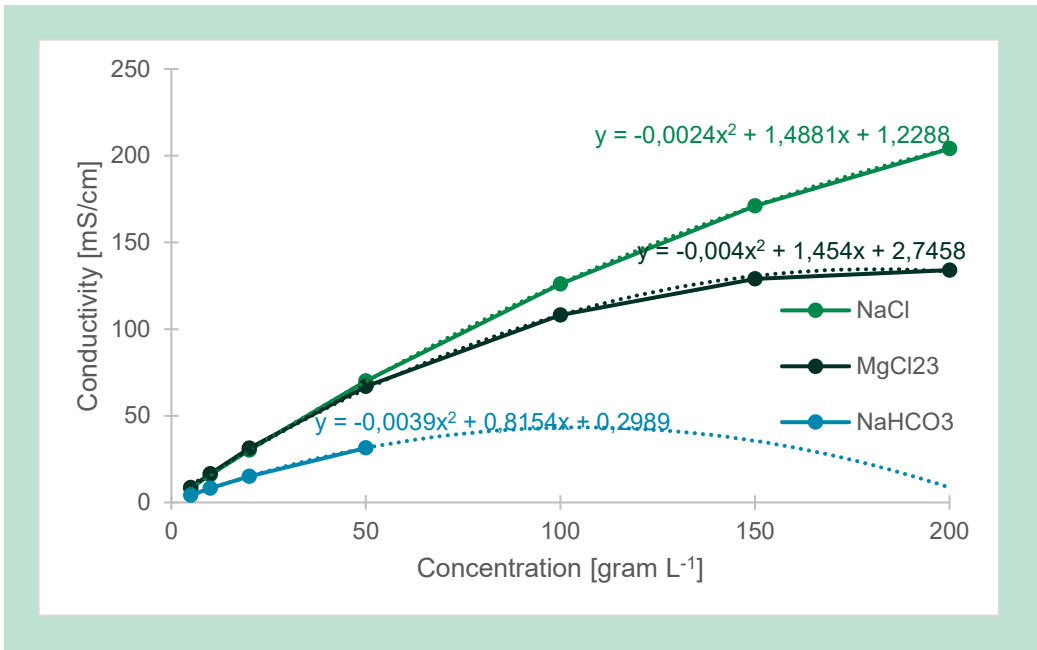
FIGURE 6. Correlations used between conductivity and salt concentrations.

The results are seen in FIGURE 7. Recovery rates between 94 % and 96 % were obtained for all treatments. Overall, the water fluxes are similar for all salts, even though results show some irregularities for the 2.8 MPa draw solution tests. As can be seen, the reverse salt flux is lower for $MgCl_2$, which would result in lower salt loss and less frequent salt refill. The relative reverse salt flux for $MgCl_2$ is around 0.1-0.2 g L^{-1} for the three tests, compared to 0.3 – 0.5 g L^{-1} for NaCl.
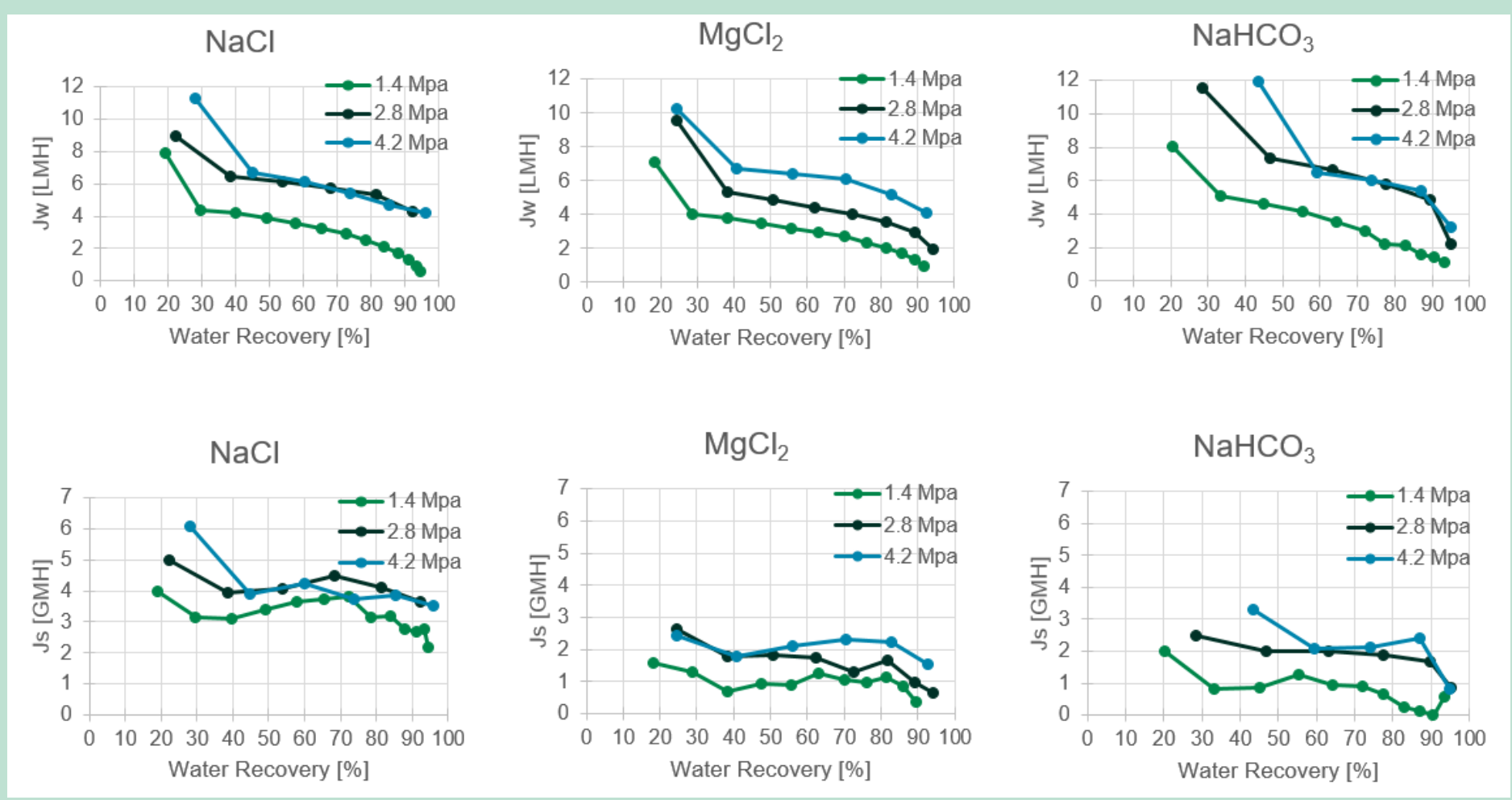
FIGURE 7. Water fluxes (LMH = L m^{-2} hour^{-1}) and Revers salt fluxes (GMH = g m^{-2} h^{-1}).

NaHCO$_3$ has limitations in solubility which is problematic. To create 4.2 MPa osmotic pressure in the draw solution, 93.3 g L^{-1} is required. At 15°C the maximum solubility is 96 g L^{-1}. As osmotic pressures higher than 4.2 MPa and temperatures below 15°C will be required in the final system, NaHCO$_3$ is not a workable alternative, and the following analysis therefore only involves NaCl and MgCl$_2$.

A big difference is in the ammonium rejection. The concentration of nitrogen in the concentrated feed, shown in FIGURE 8, is an indication of the rejection: the higher it is, the higher the rejection is, too. A high rejection is desirable, since ammonium passing to the draw solution deteriorates its quality and may pose a challenge for the final outlet quality. Ammonium rejection increases with increasing osmotic pressure for both salts, but it does so much more starkly for MgCl$_2$.

It is well-known from other studies [2] that ammonium forward flux increases with draw solute reverse salt flux, as the sodium ions facilitates active transport of ammonium ions to the draw side. Therefore, rejections are lower for NaCl compared to MgCl$_2$.
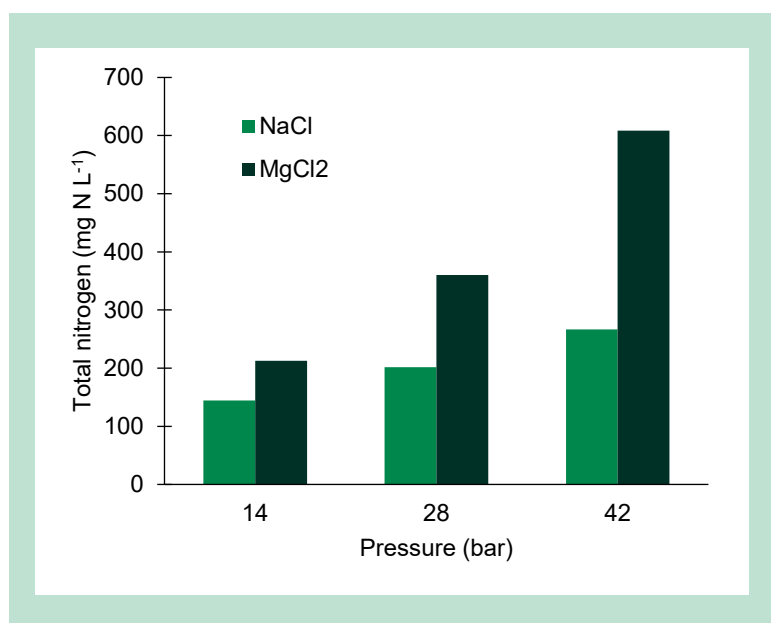
FIGURE 8. Total nitrogen concentration in the concentrated feed at different osmotic pressures.

Based on these results it looks like a sustainable flux below 4 LMH can be considered for the final system. MgCl$_2$ looks like the most attractive salt in terms of ammonium rejection. However, MgCl$_2$ comes with a higher risk of scaling, a higher level of corrosiveness and a higher power consumption (see section 5.4).

5.4   Maximum recovery flux and salt test

The above tests stopped at lower recovery rates than 99% therefore, a test was performed with the aim of getting an idea of the necessary osmotic pressure difference at the end of a full cycle (99% recovery) to maintain a flux. Considering that the feed is expected to reach 40 bar osmotic pressure, osmotic pressures of 50, 60 and 70 bar were tested. The test was performed on simulated salt solutions with similar osmolarity as the up-concentrated wastewater from Herlev Hospital following the test setup in FIGURE 9.
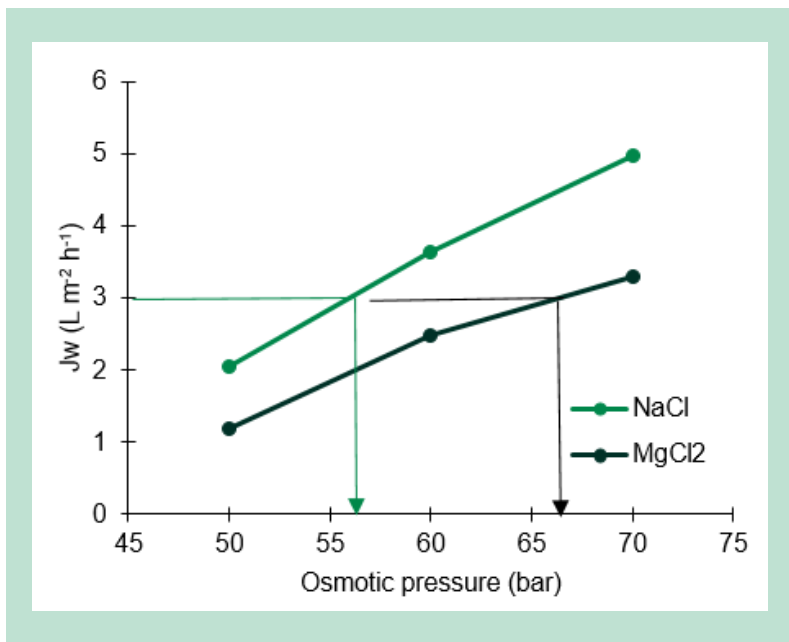
FIGURE 9. Water flux (*Jw*) as a function of osmotic pressure. The arrows indicate the osmotic pressure needed at the end of the cycle to achieve 3 L m^{-2} h^{-1}.

The results in FIGURE 9 indicate that to maintain 3 LMH, 56 bar osmotic pressure is needed using NaCl and 66 bar using MgCl$_2$ – that means 10 bar higher osmotic pressure for the same flux. This confirms that a higher power consumption is expected using MgCl$_2$ as draw solute.

5.5   Anti-biofilm agent test

The organic matter and nutrients contained in the wastewater will lead to the formation of biofilm over time on the FO membrane.

The FO membrane contains a polyamide thin film composite with integrated aquaporin proteins. The membrane is sensitive to chlorine attack and strong oxidants and therefore different types of biocide options have been evaluated in terms of their effect on membrane integrity. Membrane integrity was assessed based on two parameters: the water flux (*Jw*) and the reverse salt flux (*Js*). *Jw* refers to the net volume of water that crosses the membrane per unit area and time. *Js* refers to the net mass of salts that pass from the draw solution into the feed per unit area and time.

Overall, we do not want *Jw* and *Js* to changes over time but remain as close as possible to their original characteristics. The absolute reverse salt flux should always remain <2 g m^{-2} h^{-1} and the specific reverse salt flux (*Js*/*Jw*) should remain <0.1 g L^{-1} to keep salt losses at an acceptable level.

Initially Neuthox ® was studied to evaluate its potential as a biocide to control biofilm growth. Neuthox ® is a mixture of hypochlorous acid and hypochlorite, a very powerful disinfectant but safe in use. During operation, the system is imagined pausing feed flow and replacing it with biocide in a ready-to-use concentration. The Neuthox® manufacturing company (Danish Clean Water) recommended this procedure twice weekly with an exposure time of 1 minute to control bio growth.
A short-term test was carried out with a total of 100 min exposure time, equivalent to one year of operation, to compare two different concentrations and pH levels. The test setup was as in FIGURE 2 using Aquaporin HFFO2 FO modules.

As can be seen in FIGURE 10, 420 ppm Neuthox ® at pH 7.2 shows a better overall performance than 960 ppm Neuthox ® at pH 4.2. *Jw* remains stable over time, and *Js* is still in an acceptable range below 2 g m^{-2} h^{-1}, despite an almost doubling over time. The increased reverse salt flux is of course undesired, but 960 ppm Neuthox ® causes the *Jw* to drop to around 1/3 of the original *Jw*.

The results show two opposite effects on the polyamide layer. At pH below 7, addition of chlorine to the polyamide matrix results in a tightening of the pores and a consequent lower water flux. At pH above 7, hydrolysis by chlorine results in a less cross-linked and more hydrophilic membrane, with a subsequent higher water flux, but lower rejection rates and a higher reverse salt flux [3].
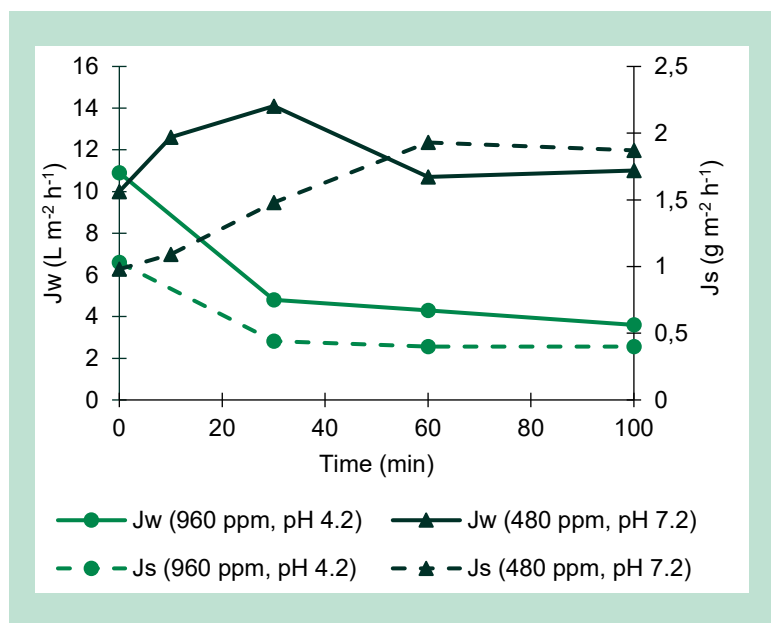
FIGURE 10. Short-term exposure test of the FO membrane to two Neuthox® solutions: 960 ppm at pH 4.2 and 420 ppm at pH 7.2. Jw is the water flux and Js is the reverse salt flux

To test further development in hydrophilicity and reverse salt flux a long-term exposure test equivalent to 9 years of operation (900 min) was performed for 420 ppm Neuthox ® at pH 7.2. The setup was as in FIGURE 2.

FIGURE 11 shows that water flux remains very stable over time, and the reverse salt flux falls back to a very low level, which is good. Thus, it was concluded that a Neuthox ® at 420 ppm and pH 7.2 is an acceptable candidate for biocide treatment.
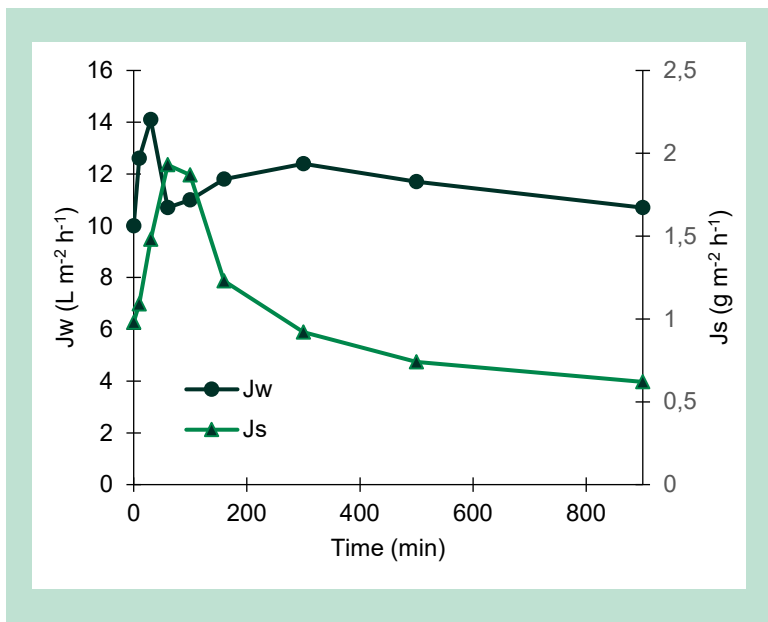
FIGURE 11. Water flux (*Jw*) and reverse salt flux (*Js)* as a function of time for Neuthox ® 420 ppm and pH 7.2.

Later other alternatives to Neuthox ® 960 ppm at pH 4.2 and 420 ppm and pH 7.2 was tested. These were:
- Neuthox ® 530 ppm and 800 ppm at pH 6.5
- Peracetic acid (PAA) 530 ppm and 650 ppm, and
- Ozone 2 ppm.

PAA 650 ppm was unstable during testing. Concentration lowered during exposure indicating reaction with the membrane, therefore the test was stopped after 120 min.
Ozone 2 ppm resulted in severe opening of the membrane pores after only 10 min of exposure time. It was not possible to test with lower concentrations (0.5 ppm intended) with the available lab equipment. However, the results indicate that ozone is a very harmful oxidant for the membrane, even at low dose.

Overall, most of the tested biocides seem to impact membrane hydrophilicity and reverse salt flux in the first 25 – 60 min of exposure and then the values stabilize. Therefore, long term testing is needed to fully see the effect of each biocide.
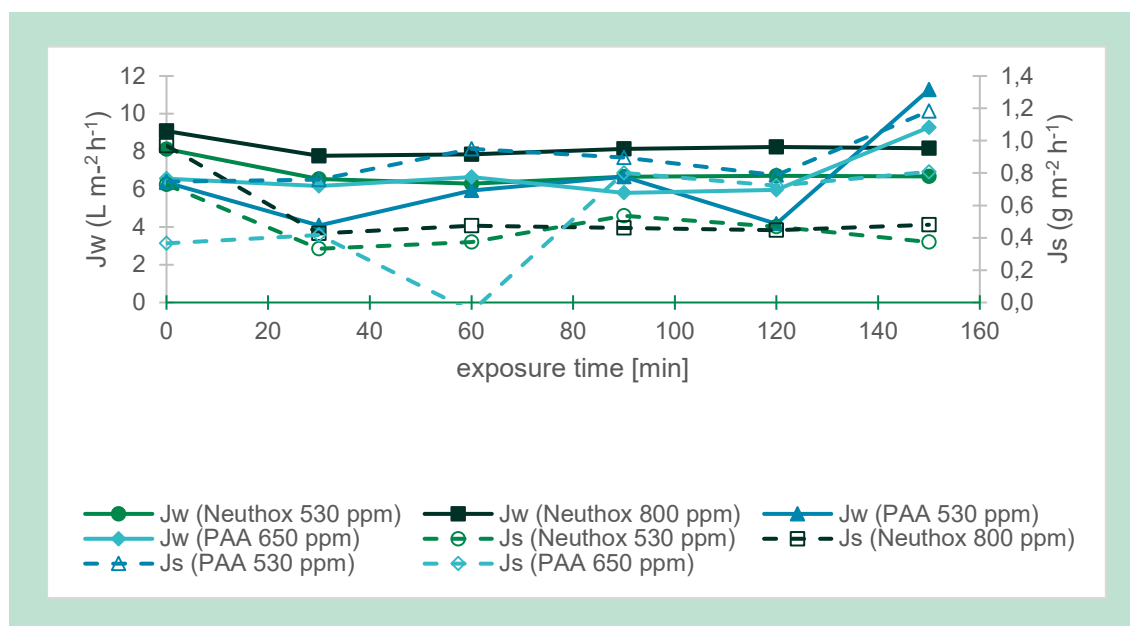
FIGURE 12. Biocide screening based on water flux (*Jw*) and reverse salt flux (*Js*).

TABLE 4. Biocide screening based on water flux (*Jw*) and reverse salt flux (*Js*).

Biocide		Neuthox 960 ppm	Neuthox 420 ppm	Neuthox 530 ppm	Neuthox 800 ppm	Peracetic acid 530 ppm	Peracetic acid 650 ppm	Ozone 2 ppm
Exposure time (min)		100	100	150	150	150	120	10
Jw (L m^{-2} h^{-1})	**Initial**	10.9	10.0	8.13	9.08	6.35	6.57	7.70
	Final	3.60	11.0	6.68	8.18	11.3	9.28	14.4
	Change (%)	-67.0	10.0	-17.8	-9.94	77.5	41.3	87.0
Js (g m^{-2} h^{-1})	**Initial**	1.03	0.98	0.73	0.97	0.75	0.37	0.73
	Final	0.40	1.87	0.37	0.48	1.18	0.81	26.8
	Change (%)	-61.2	90.8	-48.6	-50.49	58.4	121.2	3566

Both Neuthox® 530 and 800 ppm (both pH 6.5) showed very good results with almost no change in performance over time and with a specific reverse salt flux below 0.1 g L^{-1} for all test points. This looks better than Neuthox® 960 ppm (pH 4.2) which showed decreasing water flux over time and Neuthox® 420 ppm (pH 7.2) which showed very fluctuating reverse salt flux over time (increasing within the first 100 hours and then decreasing). The test results for Paracetic acid 530 ppm and 650 ppm were overall more unstable with both declines and increases in performance over time. The Paracetic 650 ppm also showed signs of reactions with the membrane during testing. Even so the specific reverse salt flux stayed below 0.2 g L^{-1} for all test points for both Paracetic acid concentrations.

Overall, the tests show that the chemicals used for biofilm control and cleaning must be carefully selected and long term tested, as the membrane seems quite sensitive towards non-appropriate cleaning chemicals.

6. Test plant design

As basis for the test plant, we decided to design the septic tank for 5 person equivalents (PE). Recommendations from the Danish Environmental Protection Agency was used for the design of the septic tank.[4] The total volume was increased to 2.7 m^3 to allow for higher loadings, so a full year could be simulated in less time without destroying settling inside the tank. Most suppliers offer septic tank volumes of 2.3 to 2.8 m^3 for a 5 PE system.

Aside from an increased volume to handle a higher loading up to 2 x 750 L/day and still allow for good settling, recommendations require the septic tank be able to accumulate a year's worth of sludge: 60 L PE^{-1} y^{-1} of floating sludge and 180 L PE^{-1} y^{-1} of settling sludge. The sum of all volumes is shown in TABLE 5.

TABLE 5. Volume allocations for the septic tank

Volume fraction	Volume (L)
Settling volume	1,500
Floating sludge volume	300
Settling sludge volume	900
Total	2,700

As basis for the design, we have used the Aquaporin HFFO14 FO membrane. With 13.8 m^2 filter area and an assumed realizable flux of up to 3 LMH, this equals 41 L/hour or 750 L in 18 hours. Test plant was prepared for later testing of other FO membranes.
Two identical parallel lines of FO-RO are installed, to be able to test different salts and make comparable studies of different designs, control philosophies and components.

A treatment capacity beyond the required 750 L/day enables simulation of a full year (274 m^3) in less than 365 days.

The characteristics of the test plant used, are summarized below. The test plant ended like this. Changes since start-up, are referred to in the text sections below and in the logbook appendix.
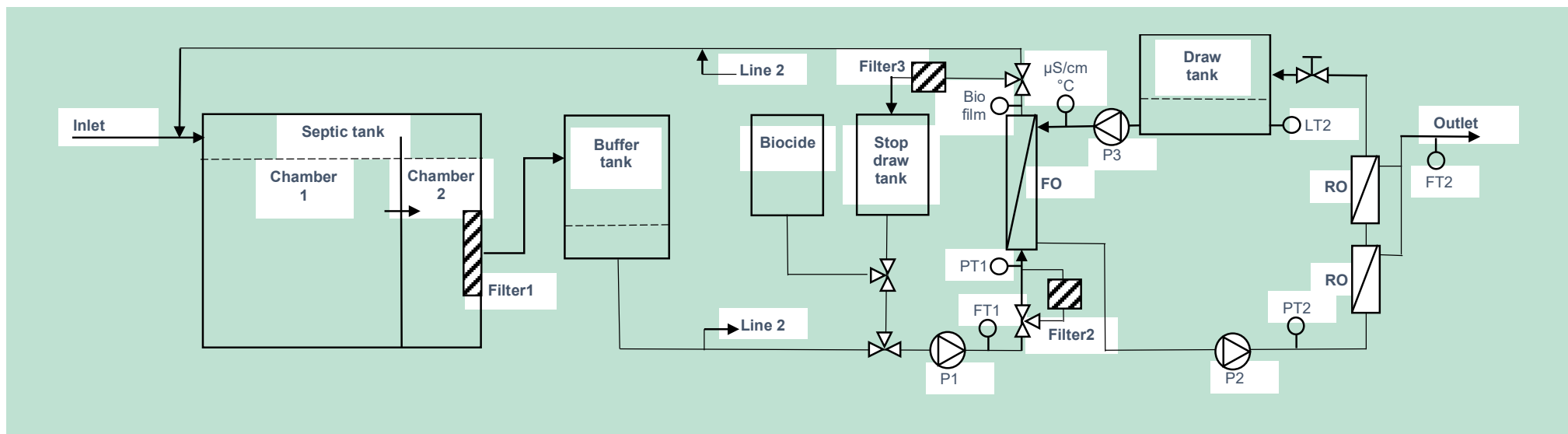
FIGURE 13. Test plant schematic and details at the end of the test period.

Inlet: Raw wastewater from Herlev Hospital taken out after macerator and inlet pumps and before fine screens.

Septic tank: 2815 L total volume (at water level 50 mm below inlet, adjustable down to 150 mm below inlet which will reduces volumes)

2025 litre in chamber 1 (72% of total) – wet volume dimensions lxbxh - 1406 x 960 x 1500 mm

791 litre in chamber 2 (28% of total) – wet volume dimensions lxbxh - 549 x 960 x 1500 mm

Surface to volume ratio = 0,67

Opening from chamber 1 to 2 in 1000 mm above bottom 50x600 mm

Filter1   Flat vertical filter - effective area bxh = 800x600 mm = 0,48 m^2 - center is 750 mm above bottom.

Buffer tank: 750 litre buffer volume

Wet volume dimensions ø1000 x 955 mm effective water height (will go down if water level is adjusted below 50 mm under inlet)

P1 pump: Impeller type - variable frequency driven with flow range up to app. 1000 litre/hour at 750 rpm.

Filter2   Cartridge filter type -10" -replaceable/rewashable

FO   Forward Osmosis membrane. Hollow fiber Inside out filtration type.

Stop draw tank: 25 litre gross volume, can be filled with tap water, permeate water or salt water.

Filter3   Cartridge filter type -10" – replaceable/rewashable

Biocide: Biocide and/or membrane cleaning chemical supplied ready-to-use in 25 litre canisters.

Biofilm sensor: Showing level of biofilm (1-100% coverage) on the sensor head.

Draw tank: 296 litre tank, unpressurized, wet volume dimensions lxbxh – 830 x 545 x 655 mm for balancing and refilling of draw solution salt.

P3 pump: Circulator type pump to ensure sufficient inlet pressure to the high-pressure pump.

P2 pump: High pressure pump. Positive displacement type. Max flow = 0,00407 litre/rev x 1500 rpm = 366 litre/hour. Max pressure 83 bar(g).

RO membrane: Applied membranes type SW30-2521. Seawater type. 2,5" diameter and 21" long. Max operating pressure 69 bar(g) (1000 psi)

7. System performance

7.1   Pre-treatment

Pre-treatment constitutes treating the raw influent and bringing it to the feed side of the FO membrane in a robust and cost-effective way that supports the overall targets for the product.

7.1.1   Septic tank

First step of pre-treatment is a standard septic tank, as the final product must be able to work with the outlet from such septic tanks. The septic tank used for testing, consist of settling in the septic tank and a proposed filtration through a vertical filter between the septic tank and the buffer tank (Filter1). The septic tank has two chambers with a baffle to promote settling. The solids in the incoming raw wastewater will settle to the bottom, the fats and oils will develop into a foam layer at the surface, and in between will be the liquid fraction to be treated. Between the two chambers is a wall with an opening to let the liquid fraction through to the second chamber. Furthermore, Filter1 is placed in such a way that only the liquid fraction of the septic tank flows through it, and so that neither the foams at the top nor the solids at the bottom meet it.

To verify the effectiveness of the septic tank, samplings have been taken using a simple suction pump with a ø10/8 mm hose and analysed for their particle size distribution.
FIGURE 14 shows an arrangement drawing of the septic tank and the sampling point for the particle size distribution shown in FIGURE 15.
FIGURE 15 shows the particle size distribution in the raw wastewater inlet and in the septic tank in chamber 1. The inlet contains particles up to 0.7 mm in diameter, while the liquid fraction after settling contains suspended particles only up to 0.3 mm in diameter. This shows that good settling takes place in the septic tank, and that it is working as intended.
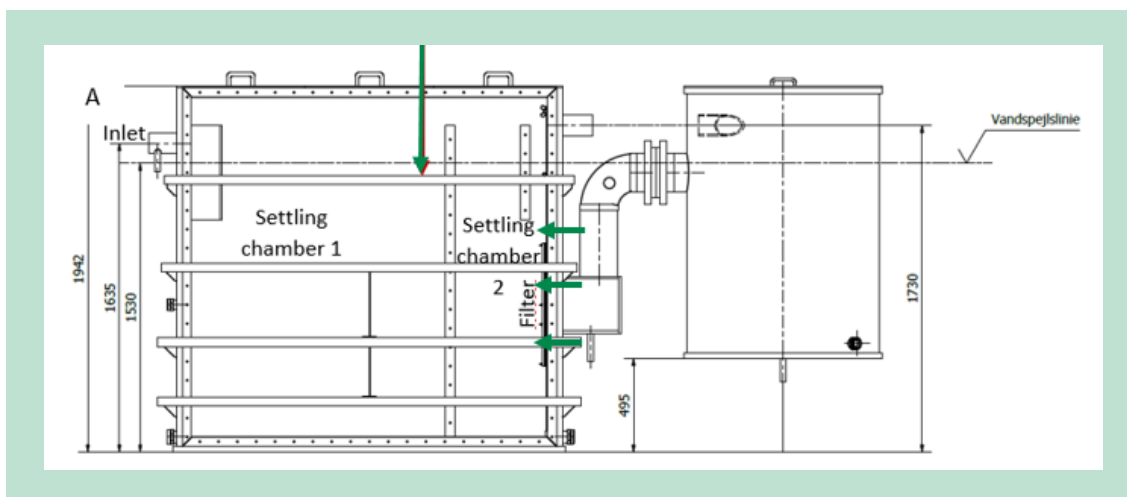
FIGURE 14. Sampling location in septic tank.
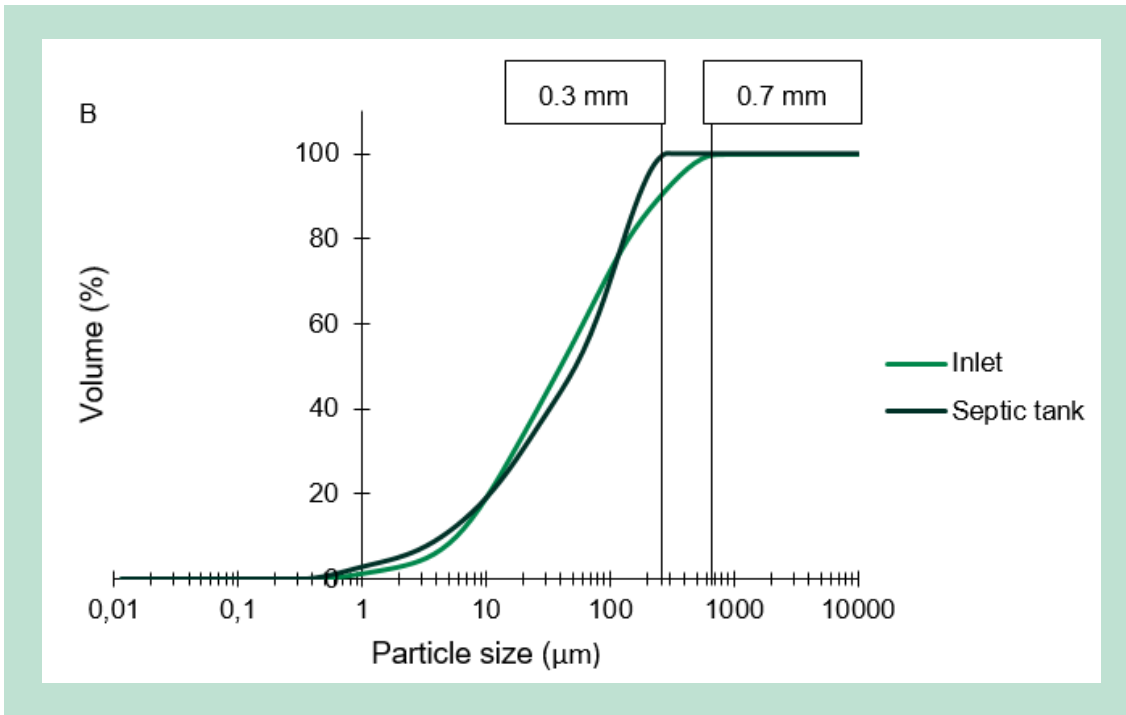
FIGURE 15. Particle size distribution in the raw wastewater inlet and the septic tank at the sampling point.

The particle size distribution of three samples, taken at various positions in front of Filter1, are shown in FIGURE 16. Sampling points Septic 1 and 2 are similar to chamber 1 with maximum particle size around 0.3 mm, whereas Septic 3 has a much lower maximum particle size around 0.035 mm. It cannot be confirmed if the sampling point for septic 3 was at a higher level, but this is what we assume happened.
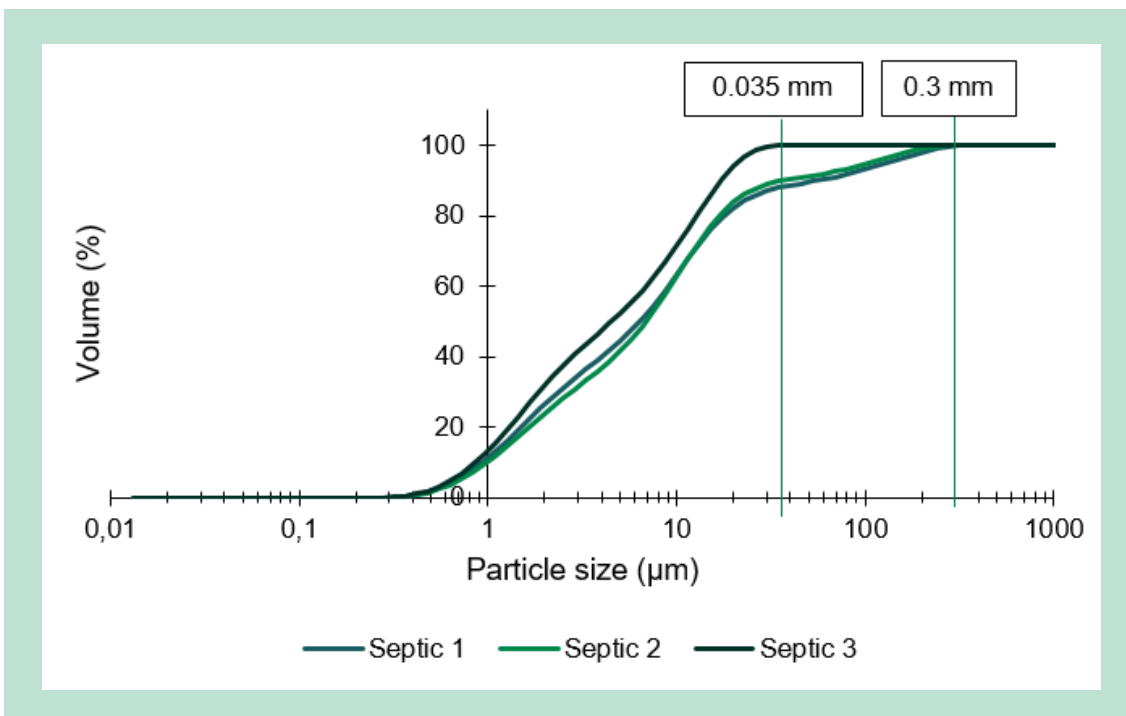
FIGURE 16. Particle size distribution at three different sampling points in the settling chamber 2 in the septic tank.

7.1.2 Filter1 – vertical filter in outlet from septic tank

The intention of Filter1 is to create a safety barrier before the FO membrane, to protect this from clogging with hair, fibres, and larger particles. To be an acceptable solution the Filter1 must be able to work 1 year between replacement intervals without any cleaning/servicing.

The four pre-filter materials in TABLE 6 were characterised according to the pressure drop across the filter and the size of the particles they let through. The reference pressure drop across the filter was 5 cm of water column since that is the recommended difference between inlet and outlet height in conventional septic tank design guidelines. A pressure drop above 5 cm is therefore considered excessive for the filter to be an acceptable solution.

TABLE 6. Pre-filter characteristics (material, pore size, type, operational conditions, operational time) and performance in terms of pressure drop over the filter immediately after installation and after a period of standstill.

Name	Fibertex F-20	Fibertex F-10	SEFAR 0.15	SEFAR 0.2
Material	Polypropylene (less permeable)	Polypropylene	Polyester	Polyester
Pore size (µm)	100 ± 30	100 ± 30	150 ± 8	200 ± 10
Type	Non-woven	Non-woven	Woven	Woven
Operation time (d)	4	9	5	284
Recirculation flow (L h⁻¹)	<2x180	<2x180	<2x180	<2x400
Number of restarts	0	1	0	Multiple
Pressure drop across filter during operation (cm)	>5	<5	>5	<5
Standstill time (d)	Discarded before standstill	7	Discarded before standstill	Multiple
Pressure drop across filter after standstill (cm WC)	-	>5	-	<5

Fibertex F-20 stood in tap water for a week while installation work was completed. It showed no anomalies in flux when commissioning started with tap water. Though signs of blockage were apparent immediately upon exposure to wastewater, and the backpressure reached 5 cm of water column within the first hour. When taken out, there were no signs of cake layer build-up on the filter, but some material seemed to have attached within the filter. This was clearly not biofilm formation either, instead we assume a combination of pore tortuosity (as it is an ir-regular, non-woven structure), material hydrophobicity and incompatibility between the zeta-potential of the material and that of the particles in the wastewater to be the cause of the clog-ging.

Fibertex F-10 worked well for 9 days after installation, while there was a continuous flow of wa-ter, it experienced severe clogging after having stood in wastewater for 7 days while the pilot was under maintenance. No cake-layer was observed on the filter surface, instead similar rea-sons like for Fibertex F-20 is assumed to be the cause.

SEFAR 0.15 mm showed problems with water flux decrease shortly after commissioning and as a result the driving water level exceeded 5 cm within hours. The driving water level was in-creased to max 15 cm to keep it running. Upon removal after 5 days of operation the filter was

inspected (Photo 1) and it showed an easily removable cake layer forming at its surface, but no evidence of biofouling.

SEFAR 0.2 mm filter has not shown any significant development in resistance during the 284 days it has been in operation and with longer periods (several weeks) with standstill in between. The driving water level has stayed below 5 cm, except from shorter periods with full load on both feed pumps (total flow close to 2,000 L/hour).

To further characterise the SEFAR filters, particle size distribution analysis was carried out. The results (FIGURE 16) after the 0.15 mm SEFAR pre-filter shows that around 95% of particles were smaller than 0.2 mm. For the 0.2 mm SEFAR pre-filter the results showed that 95% of particles are smaller than 0.6 mm. For both filters the largest particle size is around 1 mm. The results are slightly confusing, as the results shows that 5-20% of the particles after the filters are larger than the largest particle size measured in the septic tank (0.3 mm). This might be due to left over particles in the system from earlier operations, re-growth or re-flocculation of particles or uncertainties associated with probing and analysing.
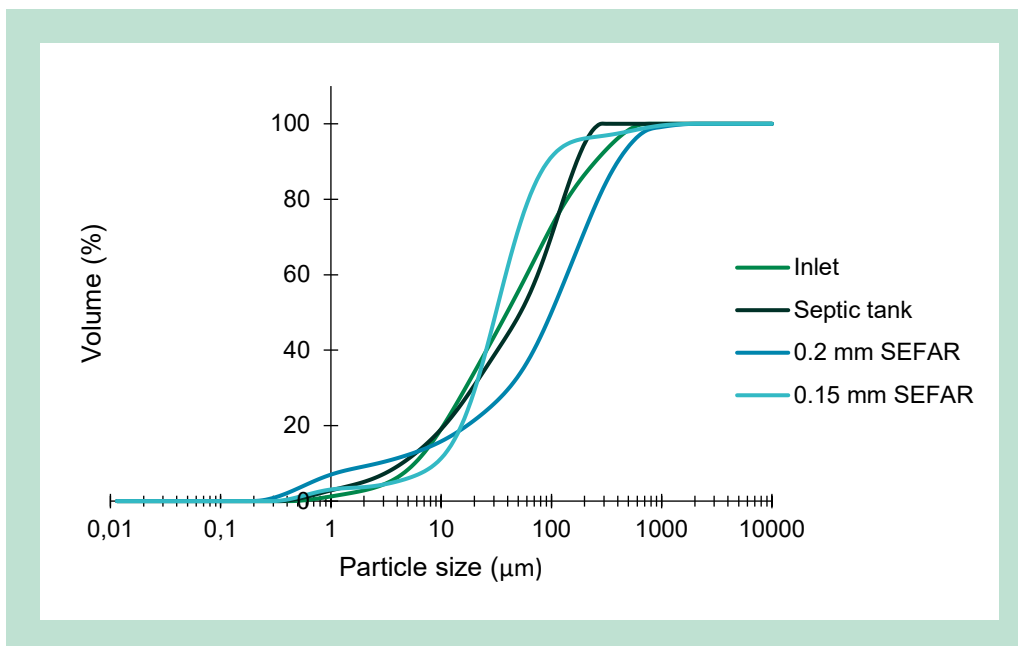
FIGURE 17. Particle size distribution of the raw wastewater at the inlet, in the septic tank and after the 0.15 mm and 0.2 mm SEFAR vertical pre-filters (Filter1).

7.1.3 TSS removal in septic tank and Filter1

The septic tank and Filter1 (SEFAR 0.2 mm) have been working well throughout the test period with good separation of heavier and lighter particles in the septic tank, so only smaller particles with a density close to water moves on to the FO membranes.

As can be seen from FIGURE 18, the influent level is averaging around 400-500 mg TSS/L, which is in line with expectations and earlier characterization of the wastewater at the hospital, which is like "thick" domestic wastewater [5]

As can be seen from FIGURE 18, the typical level for TSS after the septic tank and Filter1 is around or below 200 mg TSS/L.
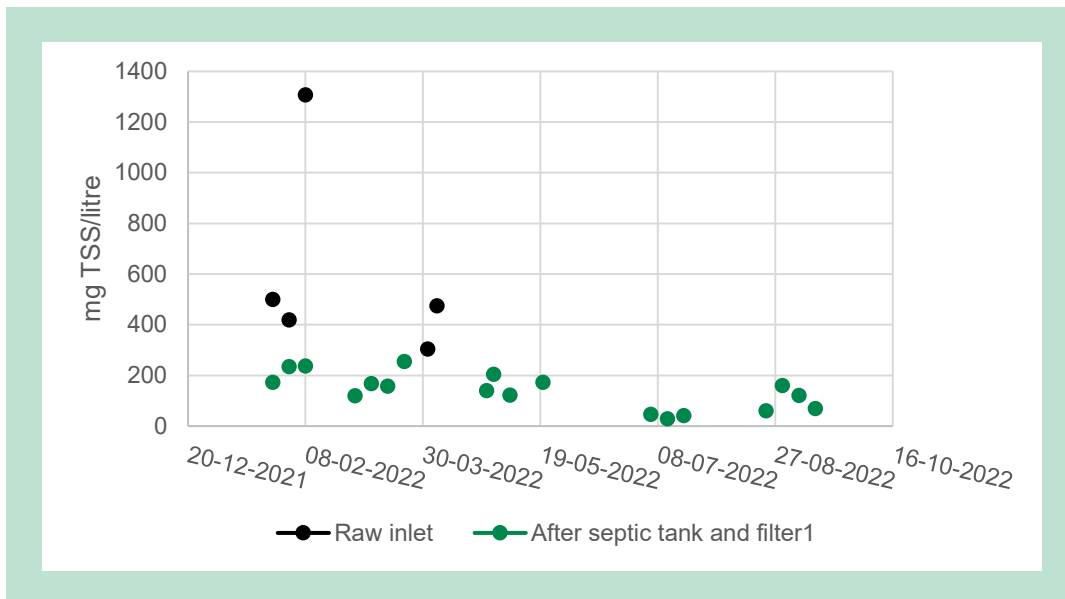
FIGURE 18. TSS in influent and after septic tank + Filter1 (SEFAR 0.2 mm).

7.1.4 Re-growth and re-flocculation test

Despite successful settling in the septic tank, there is a risk of re-flocculation and regrowth after the SEFAR 0.2 mm Filter1. Re-flocculation refers to particles aggregating as a result of collision and electrostatic interactions, and regrowth refers to bacteria forming aggregates by growing on the organic matter present in the filtered wastewater.

An experiment was carried out to assess the probability and speed of these processes. Four 300 ml bottles of wastewater sampled after Filter1 were stirred continuously at low speed to simulate conditions in the pilot plant. The bottles were incubated under anaerobic and aerobic conditions at 10°C and 25°C. The bottles were periodically sampled to determine total suspended solids and particle size distribution, and the same volume replaced with fresh filtered wastewater. Evidence of re-flocculation is expected to be visible within a few days of experiment, whereas evidence of aftergrowth is expected within a timeframe of several weeks.

As shown in FIGURE 19, aerobic conditions seem to generate an increase in total suspended solids from 0.2 to 0.5 g L^{-1}, whereas anaerobic conditions do not seem to have any significant impact.
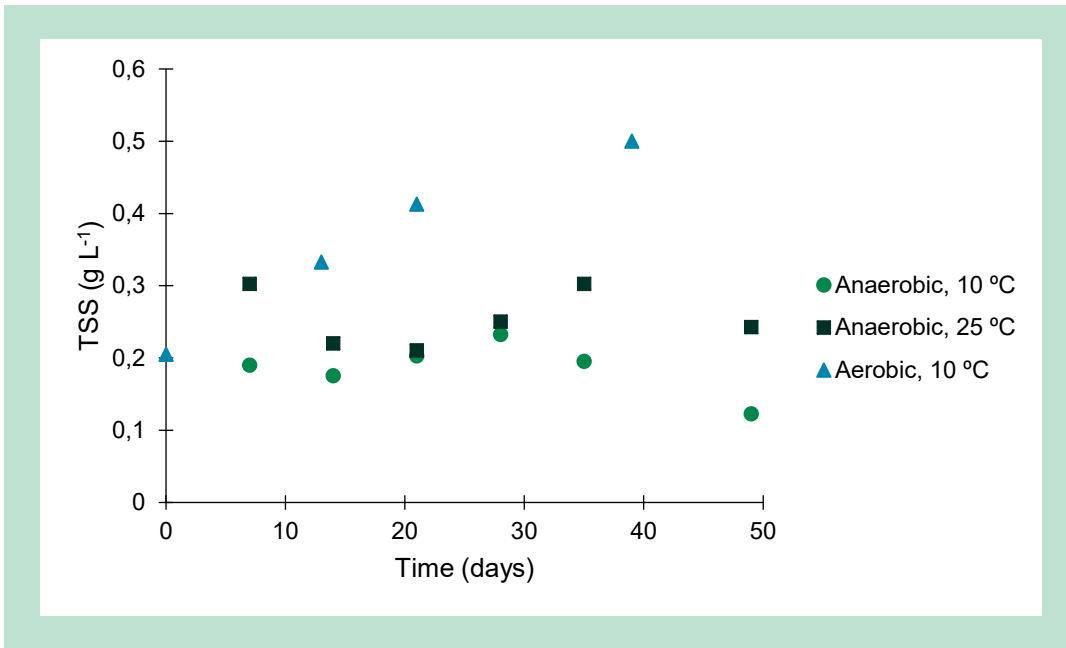
FIGURE 19. Development in total suspended solids over time.

Aggregates were observed after around 35 days of experiment (Photo 1). Yet, based on the experiments, temperature plays a less important role than oxygen to bacterial aftergrowth. FIGURE 20 shows the particle size distribution analysis for both anaerobic and aerobic conditions, which again confirms that aerobic conditions seem to generate more growth than anaerobic conditions, but also into larger particles. Higher temperature promotes also larger aggregates.

The results confirm that biological re-growth does take place after the septic tank, therefore this phenomenon must be taken into account when designing the final system.

This is further supported by visual observations done at the test plant after a long period of standstill due to maintenance works. Upon re-commissioning larger flakes of biofilm and particles was flushed out of the pipe works and captured in Filter2 and Filter3.

Aerobic/35 days/10°C

Aerobic/35 days/25°C
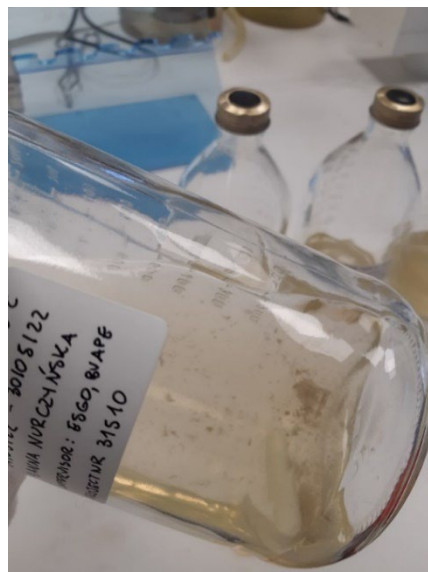
FIGURE 20. Particle size distribution analysis at the end of the regrowth experiment.

7.1.5   SEFAR 0.2 mm in combination with HFFO14

As 40% of the particles after the SEFAR 0.2 mm Filter1 is larger than 0.2 mm it was no sur-prise that this was insufficient to protect the HFFO14 with 0.2 mm hollow fibres from clogging (Photo 3). As the 0.2 mm hollow fibre FO membrane was the only one available for testing at the time, it was thus decided to implement a second pre-filtration step with smaller pore-size cartridge filters to enable further testing (Filter2).

Different micron grades of cartridge filters were evaluated. Between the 50, 25 and 10 μm car-tridge pre-filters evaluated (FIGURE 21) and tested at the test plant, it was concluded that the 50 μm filter could not do the job, as the largest particles still exceeded 0.2 mm and clogging was

not prevented. Both the 10 µm and 25 µm filters seemed to be able to protect the HFFO14 FO membrane against clogging with the largest particle size around 0.07 mm. Though as both filters remove around 70% of the particles after the SEFAR 0.2 mm Filter1 they required very frequent replacements (few hours) to work.

Such fine pre-filtration would require additional equipment and automation of some kind to be practically feasible and thus it can be concluded that the HFFO14 module with the 0.2 mm internal diameter fibres is not adequate for the application, and that larger internal diameters will be needed.
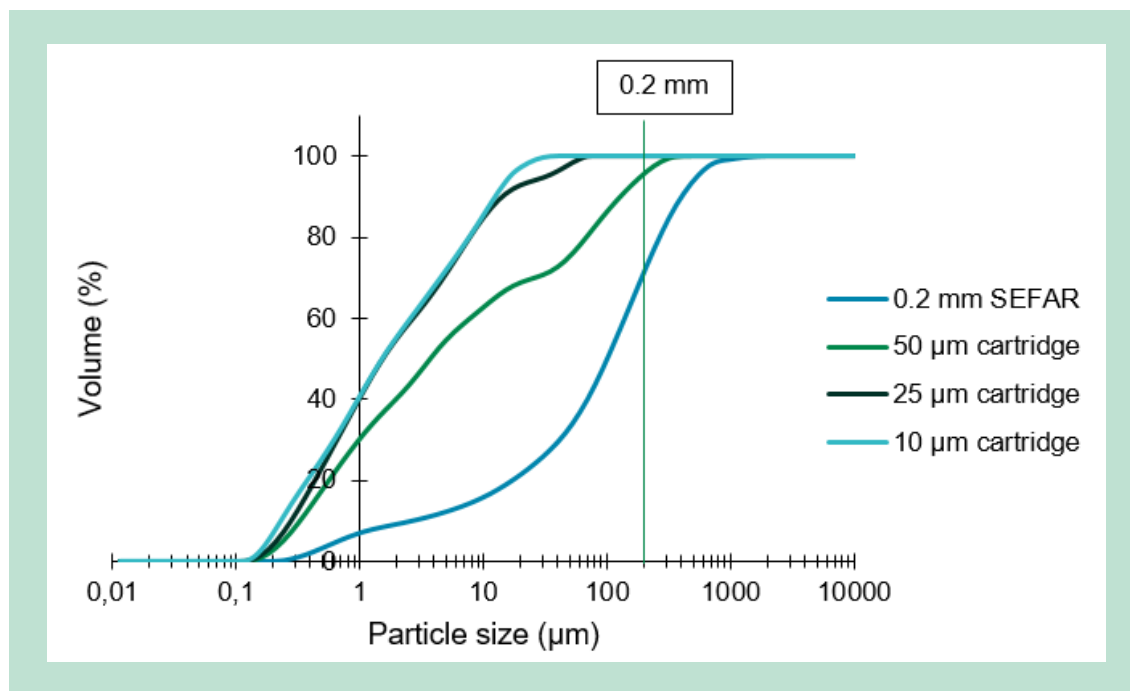
FIGURE 21. Particle size distribution for the 0.2 mm SEFAR vertical pre-filter and the 50, 25 and 10 µm cartridge pre-filters.

7.1.6 New 0.8 mm ID FO membrane

February 2022, the HFFO14 modules were substituted by the new HFFO-4065-73-31 prototype, which has an internal diameter of 0.8 mm and 6.8 m^2 membrane area.

The new FO membranes was operated for around 2 months with SEFAR 0.2 mm followed by 25-micron and later 50-micron Filter2 filtration. The operation was initially very irregular with hours of operation before stopping, due to frequent replacement of the 25-micron Filter2, later with the 50-micron Filter2, operations were more intermittent, typically with days of operation between stops. Overall, the duration of the stop periods lasted hours to days, where the membranes experienced relaxation.

Feed flows was set to 180 L/hour throughout the period for both lines and Line 1 was operated with NaCl and Line 2 with MgCl2. FO flux around 2 LMH was targeted for both lines.

Since Filter2 was changed to 70-micron, clear signs of membrane clogging emerged on Line 2 days later and two weeks later on Line 1, as the feed flows could no longer be maintained due to increased back pressure. Due to lack of sensor signals the precise back pressure development over the FO membranes cannot be told.

When stopped for cleanings, the FO feed pressure was around 2 bar on Line 2 and 1 bar on Line 1.

7.1.7 Relaxation and FO flux influence on cake layer

Late August 2022, a re-positioning of the PT1 pressure sensors from before to after Filter2 allowed a closely monitoring of the head loss development over the FO membranes during operation.

During week 35 (starting 29/8-2022), the two lines were operated with identical settings and feed flows around 400 L/hour, except from the only difference that Line 1 was operated with zero water flux and Line 2 was operated with a flux around 2 LMH.

Neither FO membranes was performing according to specifications at this time, as the reverse salt flux was extraordinarily high after intensive cleanings. Therefore Line 2 could only be operated with very frequent salt refilling and Line 1 was performing so poor, that it was automatically designated for operating without salt addition at all and zero water flux. The frequent salt refilling on Line 2 resulted in daily stops, so both lines experienced relaxation from 5–17 hours daily during this week.

As can be seen from FIGURE 22, the head loss development is very much identical on both lines at most times. This indicates that the head loss development on the FO membranes is not very dependent on whether there is water flux or not going through the membrane.

As can also be seen from FIGURE 22, relaxation has a positive impact on the back pressure on both lines as the back pressure returns to the same level after the relaxation period on both lines (around 0.35 bar for Line 1 and 0.3 bar for Line 2) even after periodically high peaks up to 0.7 bar.
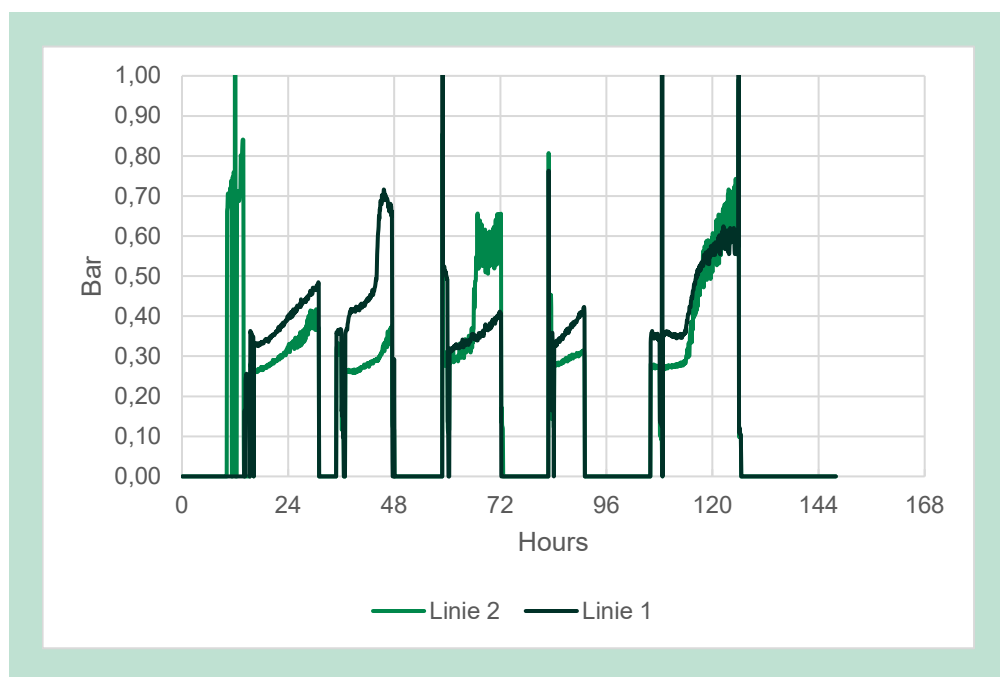
FIGURE 22. Head loss development over the FO membranes. All data points are normalized to 392 L/hour to be directly comparable as actual flow declines with increasing pressure. Hours since Monday 29/08/2022 at 00:00 hrs. Static pressure is 0.09 bar.

7.1.8 SEFAR 0.2 mm (Filter1) – 70-micron (Filter2) – 0.8 mm ID FO membrane

Since September 12, 2022, FO flux was stopped on both lines and tests was carried out to investigate the FO feed side, with the aim to find a balance between feed flow and relaxation. As can be seen from FIGURE 23 – period 14/9-17/9 - what seems to be a balance point was found at 200 L/hour feed flow and 2 x 30 min stop per day (due to a control error, this was not

100% relaxation as the pump still ran 10% (app. 100 L/hour) during stop sequence. Prior to the 30 min stop the FO membrane is filled with clean water via the stop-draw tank.
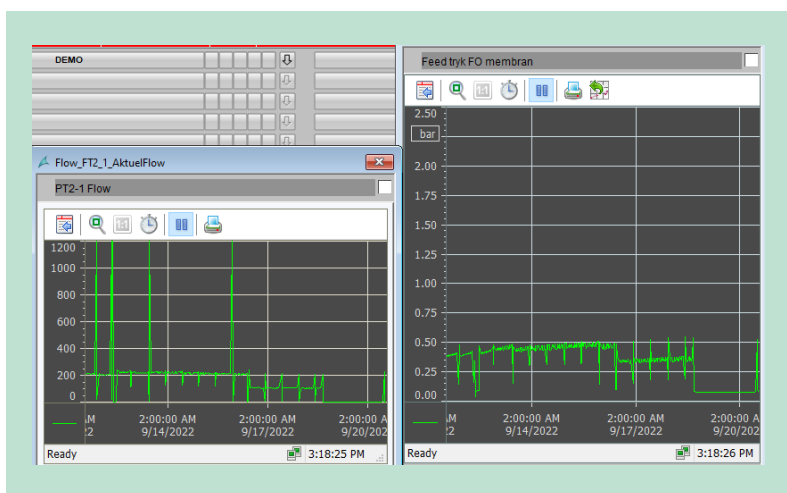
FIGURE 23. Flow and FO feed pressure on Line 1.

7.1.9   SEFAR 0.2 mm (Filter1) – 0.8 mm ID FO membrane

Following the test run above, the control error was fixed so full relaxation could be realized and the 70 my Filter2 was removed to see if a balance could be reached without Filter2. As can be seen from FIGURE 23 and FIGURE 24, the system seems to be close to balance when running at 100-120 L/hour, 2x30 min relaxation per day. Prior to each 30 min relaxation the FO membrane is filled with clean water via the stop-draw tank.
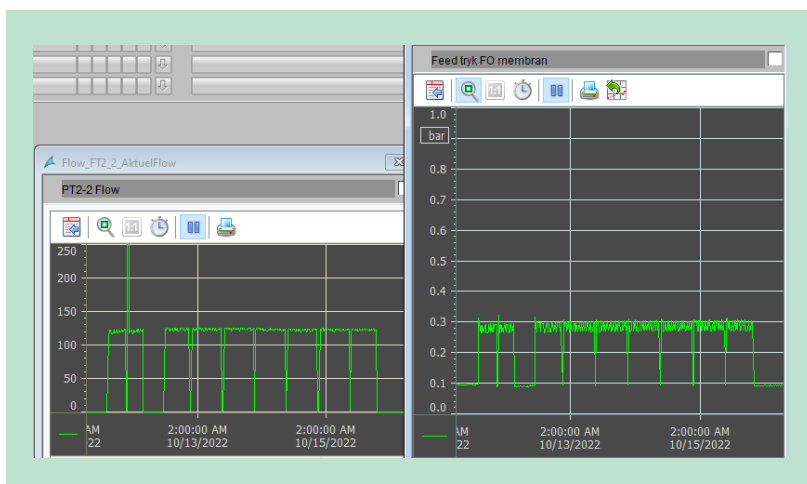
FIGURE 24. Flow and FO feed pressure on Line 2.

7.1.10   Head loss issues

To examine the head loss development more in details, theoretical calculations have been made and compared with actual developments on the test plant (FIGURE 22).

FIGURE 25 provides a schematic drawing of the feed flow side of the 0.8 mm ID FO membrane, which has been used for the head loss calculations.
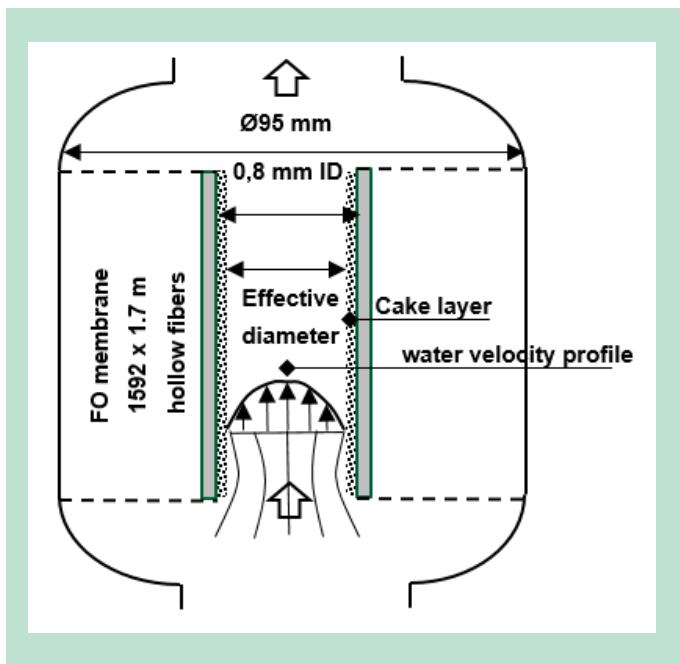
FIGURE 25. Schematic drawing of the feed side of the 0.8 mm ID FO membranes.

The head loss calculations are shown in Appendix A for three different scenarios.
Scenario definitions and results are as follows:

1. A clean FO membrane. The head loss is 0.09 bar at 400 L hour[-1]. The static pressure at point of measurement is 0.09, so the reading in FIGURE 22 will be 0.18 bar.
2. Simulation of an additional cake layer on the membrane calculated as a reduction of the internal effective diameter of the hollow fibers. To reach 0.3 bar in FIGURE 22 (head loss and static pressure) the HF internal diameter is reduced to 0.65 mm.
3. Simulation of a partly blocked membrane calculated as a fraction of completely blocked hollow fibers. To reach 0.3 bar in FIGURE 22, (head loss and static pressure) 56% of all hollow fibers must be completely blocked (and the rest fully open).

As can be seen from the calculations in Appendix A, the up flow inside the hollow fibers is clearly laminar and the head loss through the 1.7 m hollow fiber is by far the biggest head loss, whereas the inlet and outlet are only of minor significance.
The laminar flow conditions mean that there is very little shear force / up flow velocity to re-move sediment/particles from the hollow fibers and this can help explain why sediment settle inside the hollow fibers.

As the majority of the head loss is inside the hollow fibers, a good and even distribution of the water flow between all the hollow fibers is to be expected. Scenario 2 and 3 shows that it takes either 56% completely blocked hollow fibers or a 0.15 mm reduction of the hollow fiber internal diameter reduction to reach the 0.3 bar start level in FIGURE 22. Based on this, it seems rea-sonable to believe, that cake layer formations, and not so much completely blocked hollow fi-bers, are the dominant phenomenon behind the FO module head loss increase.

This will be the case as long as particles fed to the membrane are smaller than the remaining effective free gab inside the hollow fibers. This might be the explanation behind the sudden head loss peaks in FIGURE 22.
Based on the calculation model behind Appendix A, the head losses resulting from varying cake layers and feed flows has been calculated (FIGURE 26) for the FO module. As can be seen the head loss growths exponentially with increasing cake layer inside the FO membrane hollow fibers.
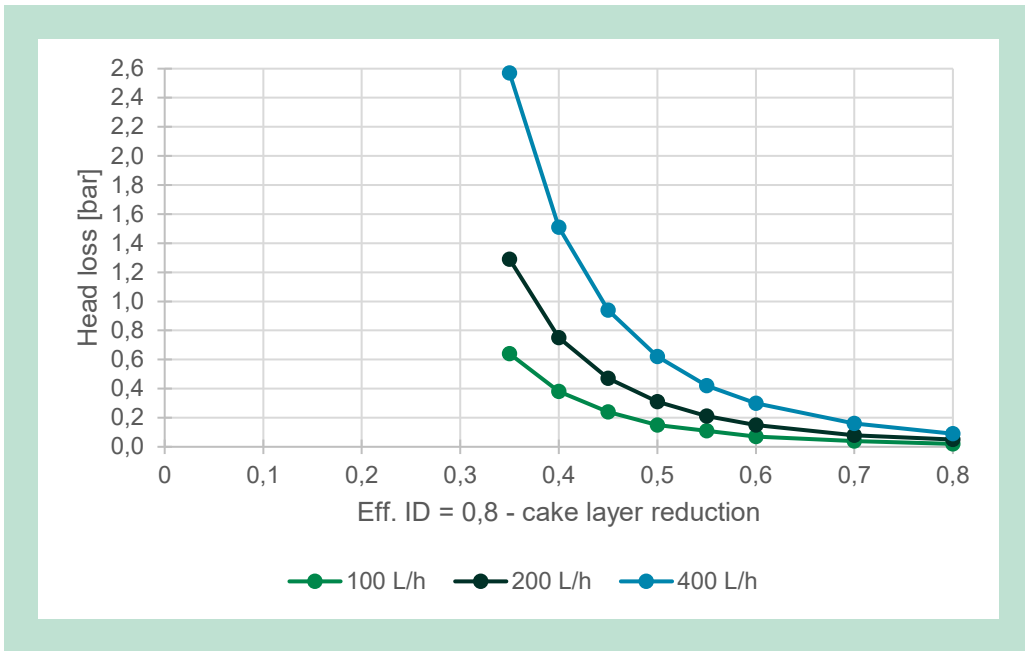
FIGURE 26. Calculated corresponding head loss and effective Internal Diameter for the 0.8 mm ID FO membrane at different feed flows.

7.1.11   Sediment inside FO membranes

On the 10/8 upon re-commissioning the FO membranes after intensive cleaning (hot water, al-kaline and citric acid cleanings) a large amount of sediment left the FO membrane and was captured in Filter3 on the return line to the stop draw tank.

As can be seen from Photo 4, the sediment clearly originates from inside the FO hollow fibers due to its long slim nature. A visual judgement estimated the collected amount of sediment to around 1 deciliter in volume (covered 2-3 cm from the bottom in the Filter3 – filter housing – picture Photo 4 furthest to the right).

The sediment was later analyzed in the lab and the content was purely organic, which supports that the cake layer formation is caused by TSS flowing to the membrane and not from scaling/precipitations caused by reverse salt flux into the feed side.

Photo 4
Sediment from 0.8 mm ID FO membranes.

7.1.12 Learnings and recommendations

From the tests and works done, we have learned that:

The septic tank works really well in securing an effluent quality without hair, fibers, fats, and oils. In addition, suspended solid concentrations are reduced efficiently and remaining particles are below 0.3 mm in size. Test results suggest that the septic tank performance can be optimized even further if the outlet is placed higher in the tank.

The SEFAR 0.2 mm can work as a filter in the septic tank outlet, though it does not have any effect in terms of reduction of suspended solids nor particle sizes. Regrowth do appear in the pipes and tanks, so the best way to employ any filter, will be closest possible to the FO membrane inlet, to act as a safety barrier against occasionally bigger particles from inlet or regrowth in the system. Smaller pore size filters 0.1-0.15 mm in varied materials and structure (woven and non-woven) was blocked very fast. Observations done and other studies [6] suggest that lack of hydrophilicity and zeta potential issues between filter and media are more likely explanations behind these problems than size exclusion effects.

The flow inside the FO hollow fibers is clearly laminar type, which combined with a high head loss in the hollow fibers contributes to cake layer build-up as long as feed flow with particles are added to the membrane. FO flux in the range below 2 LMH seems to have minor influence on cake layer formations.

Relaxation do have very good effect on cake layer reduction.
A possible explanation for this might be developed based on other studies (the critical zeta potential … and Impact of sodium hypochlorite … by biomimetic forward osmosis membranes). From these we estimate that the Aquaporin membrane is anionic with a zeta potential in the -50 to -80 mV range at pH 7-9 where we operate and that the critical zeta potential for an anionic membrane is around -40 mV. The zeta potential of the membrane goes towards zero as the salt concentration in the feed liquid increases, until it reaches the critical zeta potential and the electrostatic repulsion effect between membrane and fouling particles disappear. At this point membrane fouling will start and particles will settle on the membrane surface as cake layer. Once the feed is replaced by water, the electrostatic repulsion effect re-appears and starts "pushing" off the cake layer again.

A balance point between feed flow and relaxation on the 0.8 mm ID FO membrane seems to be around 100 L/hour feed flow and 2x30 min relaxation per day after septic tank and SEFAR 0.2 mm (around 200 mg TSS/L and particle sizes below 0.3 mm).

7.2 Draw circuit FO-RO

As working principle, the FO and RO membrane are interconnected, so the draw solution recirculates directly from the FO membrane to the RO membrane, in order to simplify the design of the system. The connected draw tank will balance differences in FO and RO flows, until the FO flow will be equal to the RO flow. When the RO flow is higher than the FO flow, the draw tank level will go down until the salt concentration/osmotic pressure is high enough to raise the FO flow to the same level and vice versa.

7.2.1 Impressions of operation #1

To give impressions about the combined FO-RO operations we will present data from a 12-hour period from 7-8 March 2022 on Line 1. At this time the new HFFO-4065-73-31 prototypes with 0.8 mm ID and 6.8 m2 membrane area had been in intermittent operation for two weeks. Line 1 is working with NaCl.
As can be seen from FIGURE 27, the RO operating pressure (PT2) is constant during the period with 22 bar, whereas the outlet flow (FT2) declines as conductivity increases. At the same time, the draw tank level (LT2) declines, indicating that the RO outflow is higher than the FO

inflow. From the three graphs (conductivity, outlet flow and draw tank level) we see they are all still trending towards a constant level, indicating that the whole system has still not completely reached a state of equilibrium after 12 hours of operation.

This has three main reasons. 1) the *starting point* in terms of osmotic pressure/salt concentration in the draw solution was *below* state of equilibrium, 2) the *loss of salt* was extraordinarily *high* – see explanation below and 3) lack of automation for salt addition and outflow control.

As the start salt concentration is too low, the draw tank level starts to decline to increase the salt concentration. This has a negative impact on the RO outflow, that will go down as the salt concentration increases – which slows down the process against equilibrium. In addition to this, the continuously high loss of salt through the membranes will further slowdown the process.
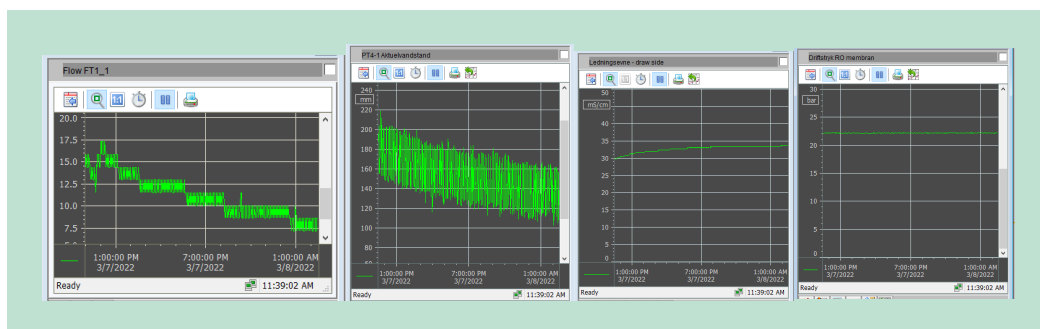
FIGURE 27. Screen dumps showing Outlet flow, Draw tank level, conductivity, and RO pressure during the period from 7-8 of March 2022 on Line 1.

Despite the system has not reached a state of equilibrium, the data can still be used to give insight into system behavior. See data from the beginning and end of the 12-hour period in TABLE 7 below:

TABLE 7. Logged and calculated data from start and end of the 12-hour period.

Time of data		07-03-2022	08-03-2022
		13:00 hrs	01:00 hrs
Feed characteristics			
Osmolarity	osmol/kg	0.247	0.247
Osmotic pressure	bar	6.1	6.1
Feed flow	L/hour	140	160
Draw solution characteristics			
Draw salt		NaCl	NaCl
i (N of ions)	Pcs.	2	2
R	L·bar/(K·mol)	0.083	0.083
Mol Weight	g/mol	58.44	58.44
Conductivity to concentration factor		0.6769	0.6769
Conductivity	mS/cm	31.2	33.4
Temperature	°C	22.5	23
Osmotic pressure	Bar	17.73	19.01
Osmolarity	osmol/kg	0.72	0.77
Molarity	mol	0.36	0.39
NaCl concentration	g/L	21.1	22.6
NaCl concentration	%	2.1%	2.3%
Height in draw tank	mm	180	137.5

Volume in draw tank	liter	81	62
NaCl content in draw tank	gram	1711	1399
RO operating pressure	bar	22	22
Draw recirculation flow/RO feed	L/hour	293	293
Outlet flow	L/hour	15	8

From logged data and grab samples taken out from Line 1 on 15 March between 13:30-14:30 – Table 17 section 8.2.1 the NaCl rejection rate for the RO membranes is calculated:

The conductivity in the draw solution averaged 47 mS/cm during this time, which equals 31.8 g NaCl/L in salt concentration on RO feed side.

The lab result showed a NaCl content in the outflow of (290 mg Na/L + 590 mg Chloride/L) = 0.88 g NaCl/L. Though the Chloride to Sodium mol ratio was 1.32, therefore the Chloride concentration used was adjusted to mol ratio 1 = 590/1.32 = 447 mg Chloride/L.

NaCl rejection rate for the RO membrane is then 1 – (0.29+0.447)/31.8 = 97.7%

This was lower than expected (99.4%) and later this turned out to be caused by leaking O-rings around the RO elements.

Together with the numbers in TABLE 7 this rejection rate can be used to detail the salt loss in the system. The outlet flow in liters is estimated by assuming a linear decline from 15 to 8 L/hour during the 12-hour period.

As can be seen below TABLE 8, the estimated reverse salt flux on the FO membrane is much higher than expected (<0.3 g/L) indicating that the FO membrane prototype had suffered damage. This together with the lower-than-expected rejection rate on the RO explains the extraordinarily high salt loss which impacted the balancing of the system.

TABLE 8. Salt losses in the FO-RO system.

Salt loss calculations		
NaCl loss in total	gram/hour	26
RO average flow out		11.5
RO NaCl re,jection rate		97.7%
RO NaCl loss	gram/hour	5.8
FO average flow	L/hour	9.9
FO average flux	L/m2/hour	1.5
FO NaCl loss	gram/hour	20.2
FO reverse salt flux	g/L	2.0

The data from TABLE 7 shows us, that during the 12-hour period an osmotic pressure difference around (17.73+19.01)/2-6.1=12.3 bar generated 1.5 LMH in average flux on the FO membrane, which is in line with expectations.

The excessive pressure needed on top of the osmotic pressure to run the RO membrane has been 22 - (17.73+19.01)/2 = 3.6 bar.

7.2.2 Impressions of operation #2

To give impressions about the combined FO-RO operations we also present data from a 21-hour period from 7-8 March 2022 on Line 2. At this time the new HFFO-4065-73-31 prototypes with 0.8 mm ID and 6.8 m2 membrane area had been in intermittent operation for two weeks. Line 2 is working on MgCl2.

As can be seen from FIGURE 28, this line is running in state of equilibrium in the FO-RO circuit. The feed flow (graph far left) is declining exponentially at the end of the period due to a

blocked Filter2 in front of the FO membrane. Despite this, the feed flow was enough for the FO membrane, and the FO-RO part was not affected.

The test plant was started up around 11:00 hrs on 7 March around 14:00 hrs - 3 hours later - it reached a state of equilibrium.

The big difference to the impression above, was that Line 2 in terms of salt concentration was starting *above* the state of equilibrium and then it was able to stabilize itself. Furthermore, it did not suffer from huge salt losses.
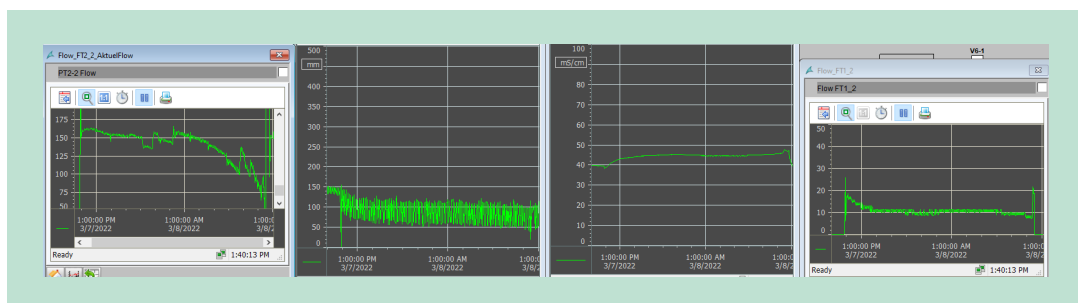
FIGURE 28. Screen dumps showing feed flow, draw tank level, conductivity, and outflow for Line 2.

See data from the beginning and end of the 21-hour period in TABLE 9 below:

TABLE 9. Logged and calculated data from start and end of the 21-hour period.

Time of sample		07-03-2022	08-03-2022
		14:00 hrs	11:00 hrs
Feed characteristics			
Osmolarity	osmol/kg	0.247	0.247
Osmotic pressure	bar	6.1	6.1
Feed flow	L/hour	160	85
Draw solution characteristics			
Draw salt		MgCl2	MgCl2
i (N of ions)	Pcs.	3	3
R	L·bar/(K·mol)	0.083	0.083
Mol Weight	g/mol	95.00	95.00
Conductivity to concentration factor		0.6168	0.6168
Conductivity	mS/cm	43.5	45.6
Temperature	°C	22.5	23.7
Osmotic pressure	Bar	20.78	21.87
Osmolarity	osmol/kg	0.85	0.89
Molarity	mol	0.28	0.30
MgCl2 concentration	g/L	26.8	28.1
MgCl2 concentration	%	2.7%	2.8%
Height in draw tank	mm	90	76
Volume in draw tank	liter	41	34
MgCl2 content in draw tank	gram	1087	962
RO operating pressure	bar	29.2	29.3
Draw recirculation flow	L/hour	293	293
Outlet flow	L/hour	12.5	9.5

From logged data and grab samples taken out from Line 2 on 15 march between 13:30-14:30 – Table 17, section 8.2.1. the MgCl2 rejection rate for the RO membranes is calculated:
The conductivity in the draw solution averaged 60 mS/cm during this time, which equals 37.1g MgCl2/L in salt concentration on RO feed side.

The lab result showed a MgCl2 content in the outflow of (1,800 mg Chloride/L + 160 mg Magnesium/L). Though the Chloride to Magnesium mol ratio was 7.7, therefore the Chloride concentration was adjusted to mol ratio 2 = 467 mg Chloride/L.
MgCl2 rejection rate for the RO membrane is then 1 – (0.16+0.467)/37.1 = 98.3%
This was lower than expected (99.4%) and later this turned out to be caused by leaking O-rings around the RO elements.

In addition, the lab results above showed remains of NaCl in the outlet pipe system from previous test phase, which caused the high mol ratio.

This rejection rate can together with the numbers in TABLE 9 be used to detail the salt loss in the system. The outlet flow in liters is estimated by assuming a linear decline from 12.5 to 9.5 L/hour during the 21-hour period.

As can be seen below TABLE 10, the estimated reverse salt flux on the FO membrane is very good and in line with expectations (<0.3 g/L).

TABLE 10. Salt loss calculations on Line 2.

Salt loss calculations		
MgCl2 loss in total	gram/hour	6
RO average flow out	L/hour	11
RO MgCl2 rejection rate		98.3%
RO MgCl2 loss	gram/hour	5.1
FO average flow	L/hour	10.7
FO average flux	L/m2/hour	1.6
FO MgCl2 loss	gram/hour	0.8
FO reverse salt flux	g/L	0.1

The data from TABLE 9 shows us, that during the 21-hour period an osmotic pressure difference around (20.78 +21.87)/2-6.1=15.3 bar generated 1.6 LMH in average flux on the FO membrane, which is in line with expectations.

The excessive pressure needed on top of the osmotic pressure to run the RO membrane has been 29.3 - (20.78 +21.87)/2 = 7.9 bar.

Learnings and recommendations.

The direct coupling of FO and RO is possible, and the system can balance itself upon start-up. It may take up to 3 hours to balance after start to reach state of equilibrium.

As expected MgCl2 requires additional pressure to reach the same flux on both FO and RO membranes.

Salt loss levels in line with expectations has been confirmed.

A rapid declining level in the draw tank and with that failure to meet state of equilibrium, is a clear signal of too high salt losses in the system, which indicates problems with the membranes.

7.2.3   Stopping the FO process

Initially a strong salt solution was pumped in from the stop draw tank to replace the wastewater on the feed side of the FO membrane to neutralize the osmotic pressure difference and stop the process. It did stop the process, but there was also a too high loss of salt to the wastewater feed side causing an unwanted increase in osmotic pressure.

Finally, we just used tap water to stop the process. The water was recirculated over the FO membrane via the stop draw tank for 30 min. This 30-min-duration was not related to stopping the process, but to remove cake layers from the FO membrane.

As can be seen from FIGURE 29, when the draw recirculation is stopped and the feed is replaced with water (and recirculated for 30 min), some flux will still go through the FO before the process stops completely. In all, it takes about 35 min before the process stops, and during this time a total of 3.6 Ls goes through the FO membrane.
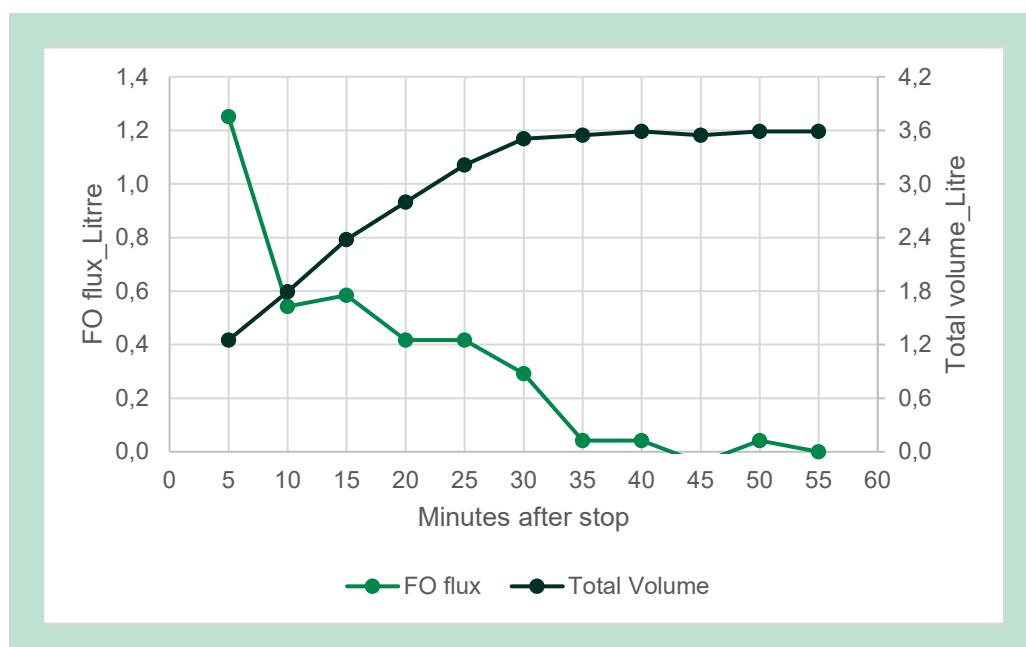
FIGURE 29. FO flow development after stop of process on the 0.8 mm ID FO membrane 6.8 m^2. $MgCl_2$ draw solution. 9 bar osmotic pressure difference at start. Zero draw flow. Feed flow for 30 min.

7.2.4   Biofilm

Biofilm will develop over time in the system, especially on the feed side of the FO membrane.

Neuthox ® was used as biocide throughout the test to control biofilm growth. The Neuthox ® was supplied as a ready-to-use mix and applied on a weekly basis, though with some longer periods without any Neuthox ® in between.

In April 2022 oxygen was detected on the FO feed side – see FIGURE 30 for system levels at one specific point in time. This is due to a combination of the *waterfall effects* in the buffer and draw tanks, where oxygen is added, recirculation flow over the septic tank and oxygen transport from draw to feed side over the FO membrane.
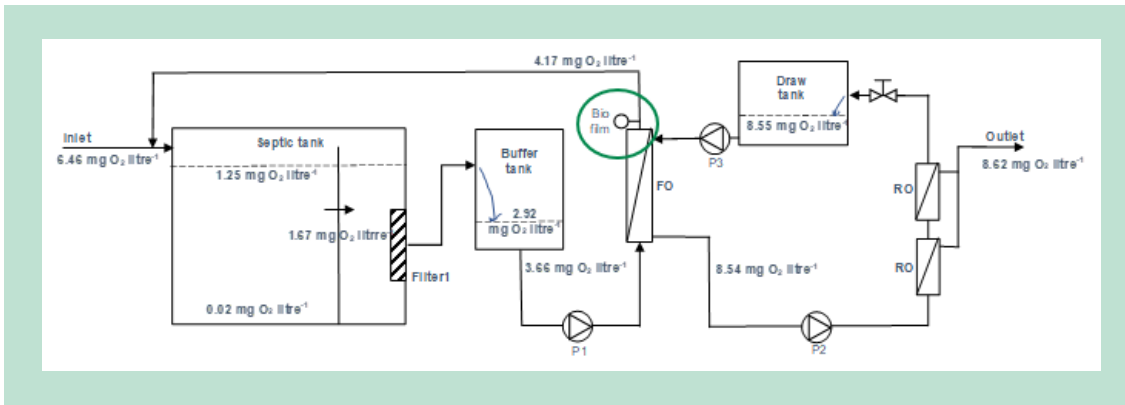
FIGURE 30. Oxygen levels with waterfall effect in draw tank 8 April 2022.

The oxygen levels in the system depends on actual operating conditions and do change over time. A high recirculation flow between septic tank, buffer tank and FO will increase oxygen levels and when recirculation flow is stopped, oxygen will be consumed, and levels will go down again.

The biofilm sensor is affected by oxygen levels in the system and by oxidizing biocide agents. The sensor signal goes from 0-100%.
After initial installation the signal reaches a base line level – this is around 50% in the system. When Neuthox®/biocide is added the signal goes towards 100%, due to oxidizing effects. When oxygen levels increase in the wastewater the biofilm sensor signal will go down. If the sensor is in open air, it reaches 0%.
When oxygen levels decrease again the biofilm sensor goes back towards baseline 50%.
When the signal increases 10-20% from "normal level" over hours/days this indicates biofilm growth in the system.

As can be derived from the biofilm sensor signal in FIGURE 31 oxygen levels have not been constant in the system but has fluctuated a lot due to changing operating conditions. Even though, it seems that the biocide treatment applied has been able to prevent biofilm from growing in the pipe system as no 10-20% peaks from baseline level is seen. Instead, the bio-cide treatment seems to have been *overdone* as the general baseline seems to lower with very frequent treatments during the second half of the 2.5-month period.
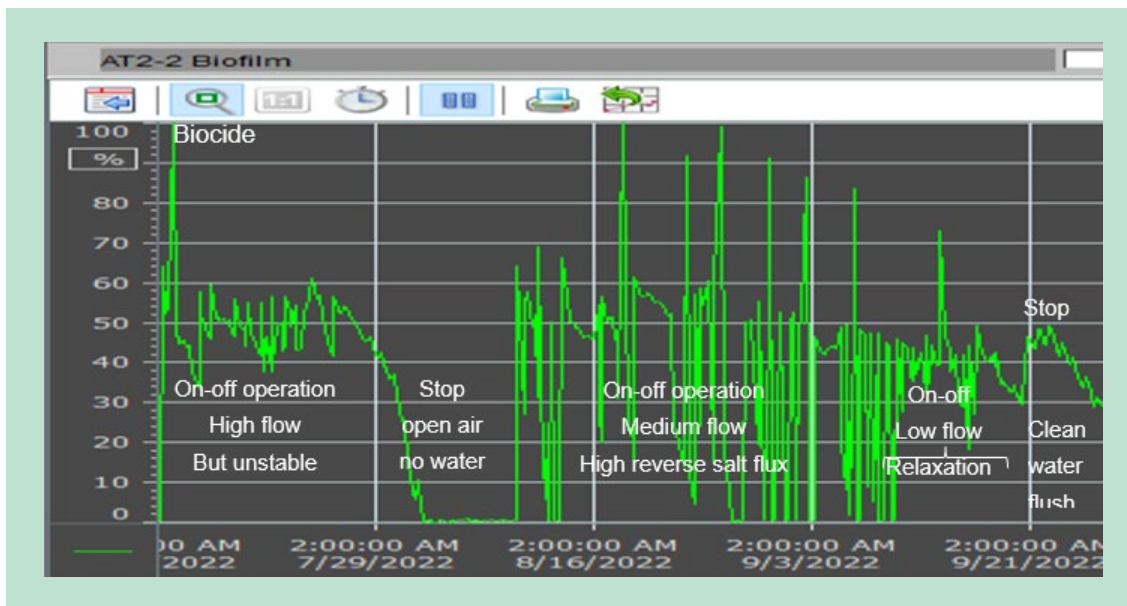
FIGURE 31. Biofilm sensors signal 11 July – 27 September 2022.

7.2.5 Hydrogen sulphide formation

During the test phase, hydrogen sulphide (H_2S) started to appear as a smell after two months of testing. As can be seen from TABLE 11, the hydrogen sulphide concentration in the buffer tank varies over time up to around 11 mg S^{2-} L^{-1} at the highest. There are also longer periods – e.g., April and May 2022 – where hydrogen sulphide was almost absent and not causing problems.

Despite high rejections in both FO and RO membrane, hydrogen sulphide was observed in draw tanks and in the final outlet with concentrations up to 0.66 mg S^{2-} L^{-1}, leading to volatisation of hydrogen sulphide into the air. The presence of hydrogen sulphide also impacts the pH value of the final outlet as it acts like a weak acid. The pH of the final outlet is typically around 8-8.5, but as the final outlet is low on alkalinity due to the combined FO and RO treatment, this caused the pH to drop to as low as 5.7. This poses both health, technical and corrosion challenges that needs to be addressed to make the system safe and fit for re-use purposes.

To fight this problem a waterfall effect was introduced in the draw recirculation loop. The *waterfall* was implemented when the draw solution returns from the RO membrane back to the draw tank (see FIGURE 13). Instead of under-water inlet, the inlet was placed around 30 cm above the surface to create a free fall and splash to aerate and drive the hydrogen sulphide out of the liquid phase. The draw tanks are open air with natural ventilation and the flow was the recirculation flow.

This solved the problem in a simple way. After a long period with no problems and no waterfall effect, hydrogen sulphide appeared again in August 2022 with 0.61 mg S^{2-} L^{-1} in the permeate. The *waterfall* was re-introduced again, which eliminated hydrogen sulphide in both draw tanks and the permeate.

TABLE 11. Hydrogen Sulphide presence and results of waterfall effect.

Date	Line / salt	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection	Remarks
		mg S_2 L^{-1}	L hour^{-1}	mg S_2 L^{-1}	mg S_2 L^{-1}	L hour^{-1}	mg S_2 L^{-1}	L hour^{-1}	% wt	% wt	
26-01-2022	L1 - NaCl	11.04		0.26			0.66				Smell - No Waterfall
	L2 - NaCl	11.04		0.21			0.18				Smell - No Waterfall
02-02-2022	L1 - NaCl	9.02		0.21			0.00				No smell - Waterfall
	L2 - NaCl	9.02		0.05			0.00				No smell - Waterfall
08-02-2022	L1 - NaCl	8.80		0.00			0.00				No smell - Waterfall
	L2 - NaCl	8.80		0.00			0.00				No smell - Waterfall
01-03-2022	L1 - NaCl	3.07		0.02			0.29				Smell - No Waterfall
	L2- MgCl$_2$	3.07		0.03			0.14				Smell - No Waterfall
08-03-2022	L1 - NaCl	1.80		0.00			0.00				No smell - No Waterfall
	L2- MgCl$_2$	1.80		0.03			0.00				No smell - No Waterfall
15-03-2022	L1 - NaCl	10.49		0.01			0.01				No smell - No Waterfall
	L2- MgCl$_2$	10.49		0.05			0.02				No smell - No Waterfall
22-03-2022	L1 - NaCl	4.98		0.01			0.01				No smell - No Waterfall
	L2- MgCl$_2$	4.98		0.03			0.03				No smell - No Waterfall
26-04-2022	L1 - NaCl	0.04		0.03			0.00				No smell - No Waterfall
	L2- MgCl$_2$	0.04		0.02			0.04				No smell - No Waterfall
10-05-2022	L1 - NaCl	0.01		0.00			0.00				No smell - No Waterfall
	L2- MgCl$_2$	0.01		0.00			0.00				No smell - No Waterfall
23-08-2022	L1 - NaCl		Stopped			Stopped		Stopped			
	L2 - NaCl	1.85	384	0.02	0.1	339	0.61	19.6	98.6%	30%	Smell - No Waterfall
30-08-2022	L1 - NaCl	1.23						Stopped			
	L2 - NaCl	1.23	369	0.02	0.0	339	0.00	11.5	99.6%	100.0%	No smell - Waterfall
06-09-2022	L1 - NaCl	0.50						Stopped			
	L2 - NaCl	0.50	308	0.03	0.0	339	0.01	8.2	101.1%	99.4%	No smell – Waterfall

7.2.6   Osmolality development

Osmolality, a measure for salt concentration, in the feed is expected to occur due to up-concentration and reverse salt flux.

According to the laboratory scale experiment, the initial osmolality of the wastewater was around 34 mOsmol kg^{-1}. After 99% feed up-concentration was reached, the osmolality of the concentrated feed was 1,650 mOsmol kg^{-1}. A similar development is expected in the pilot.

In the case of the pilot, the osmolality of the feed from the septic tank has increased more than expected, which can be seen from FIGURE 32.

The main reasons/events for this osmolality increase are assumed to be:

- Breaking of FO membranes in October 2021 caused draw solution to flow back into the septic tank.
- Using saltwater in the stop draw tanks and for stopping the FO membranes, continuously contributed with more salt to the septic tank. This was stopped February 2022.
- Longer periods of recirculating feed, but no permeate extraction or mismatch between the two will also cause a misbalance in FIGURE 18.
- Higher than expected reverse salt fluxes (higher than in the lab test) will send more salt to the septic tank.
- FO membrane failure august 2022, resulted in extraordinary high reverse salt fluxes, which explains the peaking after this point.
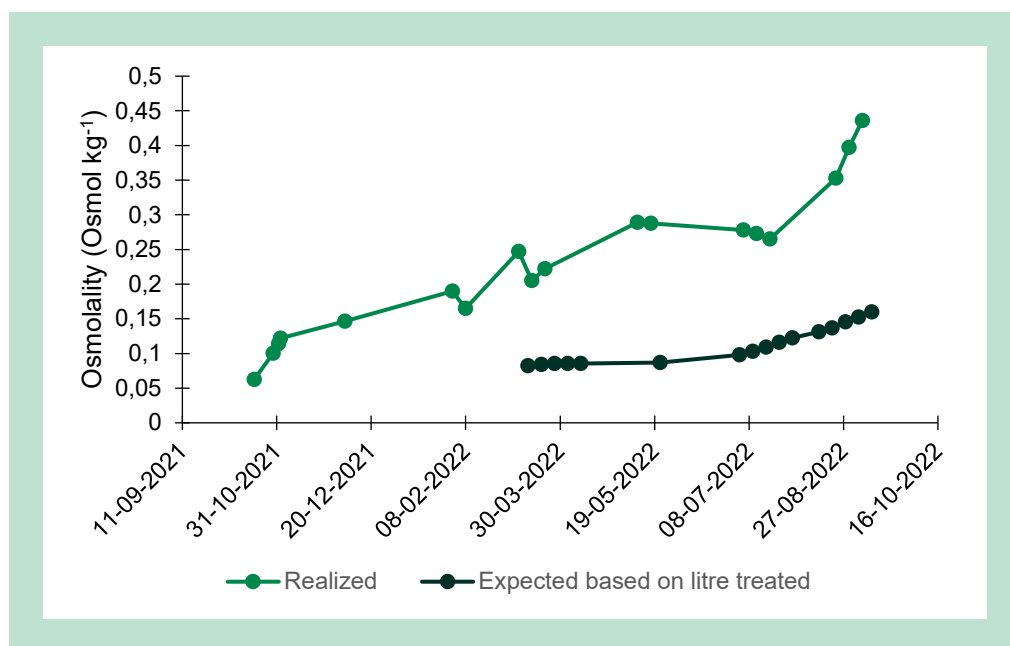
FIGURE 32. Evolution of buffer tank osmolality compared to expected due to lab test and volume treated.

8. Rejections and outlet water quality

8.1 Rejections for COD, N and P

As can be seen from TABLE 12 – 16, the membranes show overall very high rejections for both organics (COD), nitrogen and phosphorous.

The tables present operation with the 0.8 mm ID FO membrane prototype.

Rejection was calculated as follows for a time interval of 1 h:

$$\text{FO rejection} = \frac{Q_{FO,feed\ in} \cdot C_{FO,feed,in} \cdot \Delta t - [\![Q]\!]_{FO,draw\ out} \cdot C_{FO,draw\ out} - [Q_{FO,draw\ out} - Q_{FO,flux}] \cdot C_{FO,draw\ in}] \cdot \Delta t}{Q_{FO,feed\ in} \cdot C_{FO,feed,in} \cdot \Delta t} \cdot 100\%$$

$$\text{RO rejection} = \frac{Q_{FO,draw\ out} \cdot C_{FO,draw\ out} \cdot \Delta t - Q_{RO,permeate} \cdot C_{RO,permeate} \cdot \Delta t}{Q_{FO,draw\ out} \cdot C_{FO,draw\ out} \cdot \Delta t} \cdot 100\%$$

$Q_{FO,draw\ out} = Q_{recirculation\ flow}$ and $Q_{FO,flux} = Q_{RO,permeate}$ are used for calculations.

Sampling was done taking manual grab samples in the following sequence: RO permeate, FO draw out, FO draw in, FO feed in. As this method create a time gap between the samples this also add uncertainties. Uncertainties in the calculated rejections are also influenced if the system was not working in a state of equilibrium at the time of sampling, so FO flux, may not be exactly equal to RO permeate as assumed. Each sample was analysed in duplicate, and the average used for calculations. Uncertainties on lab values and flow meters also prevails. Overall, this might explain why calculations has led to >100% rejection rates in some cases.

For the period 23/8 – 6/9 the membranes were suffering from high reverse salt fluxes after intensive chemical cleanings, which might have impacted the results for this period.

Nitrogen is primarily present in ammonium (NH_4-N) form, but both Nitrate and Nitrite has been present in the test plant during periods. As described above in section 7.2.4 – Biofilm, the waterfall effect in the buffer tank and draw tank adds oxygen to the wastewater. The higher the feed flows and the more continuous operation without stops, the more oxygen is present in septic tank, buffer tank and feed side of the FO membranes, creating conditions for the development of Nitrate and Nitrite. This can be seen from TABLE 12 and 15 on Nitrate and Nitrite, with an increase in the period from 5/7 – 19/7 where recirculation was running 100% on both lines. After a long stand still period due to membrane cleanings followed by operation with only 40% on one line in the period from 23/8 – 6/9, it can also be seen that both Nitrate and Nitrite disappears again. Therefore, oxygen transfer to the septic tank must be kept low, when designing the final system, so the presence of Nitrate and Nitrite is eliminated.

Phosphorous will only be present as phosphate (PO_4) after the FO membranes, as all other forms will be part of the solids rejected by the membrane.

TABLE 12. COD rejections – measured as total COD.

Date	Line / draw	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection
		mg COD L^{-1}	L hour^{-1}	mg COD L^{-1}	mg COD L^{-1}	L hour^{-1}	mg COD L^{-1}	L hour^{-1}	% wt	% wt
12-07-2022	L1-NaCl	212	297	87	91	339	8	7.5	96.4%	99.8%
	L2-MgCl$_2$	212	713	170	164	136	22	8.0	99.6%	99.2%
19-07-2022	L1-NaCl	232	306	169	169	339	40	3.5	98.9%	99.8%
	L2-MgCl$_2$	232	657	248	244	136	Not measured	Stopped	100.4%	NA
23-08-2022	L1-NaCl	617	Stopped	Not measured	Not measured	Stopped	Not measured	Stopped	NA	NA
	L2-NaCl	617	384	697	241	339	22	19.6	159.6%	99.5%
30-08-2022	L1-NaCl	388	363	Not measured	Not measured	339	Not measured	Stopped	NA	NA
	L2-NaCl	388	369	409	490	339	34	11.5	77.5%	99.8%
06-09-2022	L1-NaCl	509	280	Not measured	Not measured	204	Not measured	Stopped	NA	NA
	L2-NaCl	509	308	492	508	339	45	8.2	93.9%	99.8%

The rejections on the FO are in general very high.

Permeate quality is below standard discharge requirements (125 mg/L) for all measurements. The permeate quality can be further optimized by increasing the recovery rate on the RO membrane from current 1-6% so the targeted discharge quality (<15 mg COD L-1) can always be met.

TABLE 13. Nitrogen rejection – measured as ammonium-based Nitrogen NH_4-N.

Date	Line / draw	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection
		mg NH_4-N L^{-1}	L $hour^{-1}$	mg NH_4-N L^{-1}	mg NH_4-N L^{-1}	L $hour^{-1}$	mg NH_4-N L^{-1}	L $hour^{-1}$	% wt	% wt
05-07-2022	L1-NaCl	32.7	750	37.4	41.6	271	6.2	5	94.6%	99.7%
	L2-MgCl$_2$	32.7	700	27.8	29.0	271	7.7	5	97.9%	99.5%
12-07-2022	L1-NaCl	24.2	297	37.0	36.5	339	2.4	7.5	98.3%	99.9%
	L2-MgCl$_2$	24.2	713	41.8	39.3	136	7.4	8.0	100.0%	98.9%
19-07-2022	L1-NaCl	6.3	306	1.2	Not measured	339	0.5	3.5	NA	NA
	L2-MgCl$_2$	6.3	657	8.7	7.9	136	Not measured	Stopped	102.5%	NA
23-08-2022	L1-NaCl	23.3	Stopped	Not measured	Not measured	Stopped	Not measured	Stopped	NA	NA
	L2-NaCl	23.3	384	51.0	52.4	339	5.3	19.6	83.4%	99.4%
30-08-2022	L1-NaCl	33.4	363	Not measured	Not measured	339	Not measured	Stopped	NA	NA
	L2-NaCl	33.4	369	59.7	59.2	339	7.3	11.5	96.0%	99.6%
06-09-2022	L1-NaCl	44.3	280	Not measured	Not measured	204	Not measured	Stopped	NA	NA
	L2-NaCl	44.3	308	81.5	72.3	339	10.1	8.2	117.9%	99.7%

Permeate quality are both below and above targeted discharge quality (<5 mg NH_4-N L^{-1}) for measurements done. Rejections rates are very high on both membranes. Though, the impact of reverse salt flux, especially when running on NaCl, is evident during the period from 23/8 – 6/9. Here the ammonium concentration in the draw tank increases much more than on the feed side, due to high reverse salt flux on the FO membrane during this period.

Addressing the reverse salt flux issue and by increasing the recovery rate on the RO membrane from current 1-6% will improve permeate quality so the discharge requirements can always be met.

TABLE 14. Nitrate rejection – measured as NO_3-N.

Date	Line / draw	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection
		mg NO_3-N L^{-1}	L hour^{-1}	mg NO_3-N L^{-1}	mg NO_3-N L^{-1}	L hour^{-1}	mg NO_3-N L^{-1}	L hour^{-1}	% mass	% mass
05-07-2022	L1-NaCl	0.07	750	0.39	0.43	271	0.13	5	75.7%	99.5%
	L2-MgCl$_2$	0.07	700	0.49	0.48	271	0.21	5	100.5%	99.2%
12-07-2022	L1-NaCl	0.19	297	0.84	0.72	339	0.09	7.5	156.5%	99.7%
	L2-MgCl$_2$	0.19	713	0.40	0.40	136	0.16	8.0	98.6%	97.7%
19-07-2022	L1-NaCl	0.41	306	2.51	Not measured	339	0.99	3.5	NA	NA
	L2-MgCl$_2$	0.41	657	0.92	1.06	136	Not measured	Stopped	93.3%	NA
23-08-2022	L1-NaCl	<0.2	Stopped	Not measured	Not measured	Stopped	Not measured	Stopped	NA	NA
	L2-NaCl	<0.2	384	<0.2	<0.2	339	<0.2	19.6	NA	NA
30-08-2022	L1-NaCl	0.05	363	Not measured	Not measured	339	Not measured	Stopped	NA	NA
	L2-NaCl	0.05	369	0.09	0.08	339	<0.2	11.5	103.9%	100.0%
06-09-2022	L1-NaCl	<0.2	280	Not measured	Not measured	204	Not measured	Stopped	NA	NA
	L2-NaCl	<0.2	308	0.16	0.17	339	0.10	8.2	NA	98.5%

Permeate quality are below drinking water requirements for all measurements (50 mg NO_3 L^{-1} equal to 12.9 mg NO3-N L^{-1}). The permeate quality can be further optimized by reducing nitrate formations on the feed side of the FO membrane as mentioned above and by increasing the recovery rate on the RO membrane from current 1-6% so the targeted discharge quality (<0.5 mg NO_3-N L^{-1}) can always be met.

TABLE 15. Nitrite rejection – measured as NO_2-N.

Date	Line / draw	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection
		mg NO_2 L^{-1}	L hour^{-1}	mg NO_2 L^{-1}	mg NO_2 L^{-1}	L hour^{-1}	mg NO_2 L^{-1}	L hour^{-1}	% mass	% mass
05-07-2022	L1-NaCl	0.5	750	4.1	4.1	271	0.9	5	94.8%	99.6%
	L2-MgCl$_2$	0.5	700	1.5	1.5	271	0.5	5	96.9%	99.4%
12-07-2022	L1-NaCl	4.3	297	17.4	17.7	339	1.2	7.5	84.5%	99.9%
	L2-MgCl$_2$	4.3	713	6.2	6.2	136	1.8	8.0	98.5%	98.3%
19-07-2022	L1-NaCl	18.2	306	22.3	Not measured	339	11.5	3.5	NA	NA
	L2-MgCl$_2$	18.2	657	16.1	15.1	136	Not measured	Stopped	101.1%	NA
23-08-2022	L1-NaCl	<0.1	Stopped	Not measured	Not measured	Stopped	Not measured	Stopped	NA	NA
	L2-NaCl	<0.1	384	<0.1	<0.1	339	<0.1	19.6	NA	NA
30-08-2022	L1-NaCl	0.2	363	Not measured	Not measured	339	Not measured	Not measured	NA	NA
	L2-NaCl	0.2	369	0.1	0.1	339	<0.1	11.5	100.1%	100.0%
06-09-2022	L1-NaCl	<0.1	280	Not measured	Not measured	204	Not measured	Not measured	NA	NA
	L2-NaCl	<0.1	308	0.7	0.7	339	0.2	8.2	NA	99.2%

Permeate quality has in longer periods exceeded drinking water requirements (0.1 mg NO_2 L^{-1} equal to 0.03 mg NO_2-N L^{-1}). This issue shall be addressed by reducing nitrite formations on the feed side of the FO membrane as mentioned above and by increasing the recovery rate on the RO membrane from current 1-6% so the targeted discharge quality (<0.02 mg NO_2-N L^{-1}) can always be met.

TABLE 16. Phosphorous rejection – measured as phosphate-based phosphorous PO$_4$-P.

Date	Line / draw	FO feed	FO feed	FO Draw in	FO Draw out	Recirculation	RO permeate	RO permeate	FO rejection	RO rejection
		mg PO$_4$-P L^{-1}	L hour^{-1}	mg PO$_4$-P L^{-1}	mg PO$_4$-P L^{-1}	L hour^{-1}	mg PO$_4$-P L^{-1}	L hour^{-1}	% mass	% mass
05-07-2022	L1-NaCl	3.5	750	1.4	1.7	271	<0.1	5	96.0%	100.0%
	L2-MgCl$_2$	3.5	700	3.2	3.5	271	<0.1	5	96.3%	100.0%
12-07-2022	L1-NaCl	4.0	297	1.7	1.8	339	<0.1	7.5	97.5%	100.0%
	L2-MgCl$_2$	4.0	713	5.2	5.0	136	<0.1	8.0	99.4%	100.0%
19-07-2022	L1-NaCl	3.1	306	0.7	Not measured	339	<0.1	3.5	NA	NA
	L2-MgCl$_2$	3.1	657	0.6	0.8	136	Not measured	Stopped	99.0%	NA
23-08-2022	L1-NaCl	6.4	Stopped	Not measured	Not measured	Stopped	Not measured	Stopped	NA	NA
	L2-NaCl	6.4	384	5.8	5.0	339	<0.1	19.6	106.5%	100.0%
30-08-2022	L1-NaCl	5.9	363	Not measured	Not measured	339	Not measured	Stopped	NA	NA
	L2-NaCl	5.9	369	2.0	1.9	339	<0.1	11.5	99.9%	100.0%
06-09-2022	L1-NaCl	5.6	280	Not measured	Not measured	204	Not measured	Stopped	NA	NA
	L2-NaCl	5.6	308	1.1	1.0	339	<0.1	8.2	101.9%	100.0%

Phosphorous rejections are very high on the FO membrane and always 100% on the RO membrane leaving the final outlet completely free from phosphorous

8.2 Outlet water quality

The final water quality at the outlet was tested in different ways.

The typical wastewater parameters like COD, Nitrogen and Phosphorous are evaluated above in section 8.1 rejections. Lab work was done at the DTU lab in Kgs. Lyngby, Denmark.

The water quality of the final outlet has also been tested according to drinking water quality standards and for PAHs in DK at Eurofins in Vejen, Denmark. Not because the outlet is intended for drinking water, but because it helps evaluating the water quality for the intended re-use purposes and infiltration into the ground. Results in TABLE 17.

Additional tests for Halogenated organics and PFAS connections has been done at the DTU lab. Results in TABLE 18.

Finally, the outlet has been tested for 108 different pharmaceuticals at Institute für Energie und Umwelttechnologie (IUTA) in Duisburg, Germany, to evaluate the systems performance on micropollutants. Results in TABLE 19.

Some mechanical challenges were present at the test plant at the time of sampling, which influenced lab results. The FO membrane integrity was OK on both lines, but it turned out that the RO membranes were leaking around the o-rings when sampling campaigns was running on the 15/3. In addition, both permeate lines were connected to hoses intended for auto sampling during sampling. These transparent hoses were filled with old RO permeate water from the beginning of the project and biological activity was visible in the hoses, which might have impacted on results as well.

8.2.1 Drinking water requirements

Some comments on the drinking water results.

The unpleasant smell might be influenced by very low hydrogen sulphide concentrations (lab results said 0.01 mg S2- L^{-1}). This can be improved with the "waterfall" effect.

Evaporation residue (TDS), conductivity, magnesium, sodium, and chloride are closely linked to RO membrane integrity. So, these results will be improved when leaks are eliminated.

The test plant RO permeate system is not optimized in terms of preventing bacterial growth and there are dead zones present where this can take place (sampling hoses as mentioned above). This can be optimized through a better system design (construction, materials, and operation/cleaning protocols) which will benefit total plate count at both 22°C and 37°C.

TABLE 17. Drinking water and heavy metals analysis (L1 – NaCl draw solution) and (L2 – MgCl$_2$ draw solution).

Component	Unit	Result Outlet L1 (15/03/22)	Outlet L2 (15/03/22)	Drinking water limit value	Method
Colour		No colour	No Colour		
Clearness		Clear	Clear		
Smell		Unpleasant	Unpleasant		
pH	pH	8.4	7.4	7-8.5	DS/EN ISO 10523

Temperature(pH)	°C	21	21		DS/EN ISO 10523
Evaporation residue	mg/l	850	530	1500	DS 204
Oxygen	mg/l	9	8.0	>5	EN 25814
Conductivity	mS/m	200	86	>30	DS/EN 27888:2003
Colour no., Pt	mg Pt/l	< 1	< 1		DS/EN ISO 7887:2012, metode C
Turbidity	FNU	0.11	< 0.07	1	DS/EN ISO 7027-1: 2016.
Coliforme bacteria	MPN/100 ml	< 2	< 2	None detect.	DS 2255:2001
Escherichia coli	MPN/100 ml	< 2	< 2	None detect.	DS 2255:2001
Plate count 22°C	CFU/ml	2200	1900	200	ISO 6222:1999
Plate count 37°C	CFU/ml	2000	1050	20	ISO 6222:1999
Total hardnesss	°dH	< 0.1	15	5-30	SM 3120 ICP-OES
Ammonium (NH_4)	mg/l	18	8.7	0.05	SM 17. udg. 4500-NH3 (H)
Nitrite	mg/l	0.0066	0.0033	0.1	SM 17. udg. 4500-NO2 (B)
Nitrate	mg/l	0.44	0.48	50	SM 17. udg. 4500-NO3 (H)
Total Phosphorous	mg/l	< 0.01	0.01	0.15	DS/EN ISO 6878:2004 part 7 + ISO 15923-1:2013
Chloride	mg/l	590	250	250	SM 17. udg. 4500-Cl (E)
Fluoride	mg/l	< 0.05	0.17	1.5	SM 17. udg. 4500-F (E)
Sulfate, filtered	mg/l	0.79	0.72	250	SM 17. udg. 4500-SO4 (E)
Aggressiv CO_2	mg/l	10	15	2	DS 236:1977
Hydrogen carbonate	mg/l	32.2	19.6		DS/EN ISO 9963
NVOC	mg/l	0.19	<0.1	4	DS/EN 1484
Calcium (Ca)	mg/l	0.6	<0.5	200	DS/EN ISO 17294m:2016 ICP-MS
Iron (Fe)	mg/l	< 0.01	< 0.01	0.2	DS/EN ISO 17294m:2016 ICP-MS
Potassium (K)	mg/l	6.8	1.9	10	DS/EN ISO 17294m:2016 ICP-MS
Mercury (Hg)	µg/l	< 0.05	< 0.05	1	DS/EN ISO 17294m:2016 ICP-MS
Magnesium (Mg)	mg/l	< 0,1	57	50	DS/EN ISO 17294m:2016 ICP-MS
Manganese (Mn)	mg/l	< 0.002	<0.002	0.05	DS/EN ISO 17294m:2016 ICP-MS
Natrium (Na)	mg/l	290	30	175	DS/EN ISO 17294m:2016 ICP-MS
Silver (Ag)	µg/l	< 0,1	< 0,1	10	DS/EN ISO 17294m:2016 ICP-MS
LAS	µg/l	< 3	< 3		M 0386 LC-MS/MS
Boron (B)	mg/l	0.12	< 0.1	1	DTU-lab
Chrome (Cr)	µg/l	< 1	< 1	50	DTU-lab
Cupper (Cu)	µg/l	< 0.05	<0.05	2000	DTU-lab
Nickel (Ni)	µg/l	1	2	20	DTU-lab
Zinc (Zn)	µg/l	< 2	< 2	100	DTU-lab
Lead (Pb)	µg/l	< 0.1	< 0.1	5	DTU-lab
Cadmium (Cd)	µg/l	< 0.1	< 0.1	3	DTU-lab
Sum 16 PAHs	µg/l	N.D.	0.11		M 0250 GC-MS
Naphthalen	µg/l	<0.01	0.092		M 0250 GC-MS
Acenaphthylen	µg/l	<0.01	<0.06		M 0250 GC-MS
Acenaphten	µg/l	<0.01	<0.01		M 0250 GC-MS
Flouren	µg/l	<0.01	0.02		M 0250 GC-MS

Compound		Concentration		
Phenanthren	µg/l	<0.01	<0.01	M 0250 GC-MS
Pyren	µg/l	<0.01	<0.01	M 0250 GC-MS
Benzo(a)anthracen	µg/l	<0.01	<0.01	M 0250 GC-MS
Chrysen/Triphenylen	µg/l	<0.01	<0.01	M 0250 GC-MS
Benzo(b+j+k)flouran-then	µg/l	<0.01	<0.01	M 0250 GC-MS
Benzo(a)pyren	µg/l	<0.01	<0.01	M 0250 GC-MS
Dibenz(a,h)anthra-cene	µg/l	<0.01	<0.01	M 0250 GC-MS
Benzo(g,h,i)perylen	µg/l	<0.01	<0.01	M 0250 GC-MS

8.2.2   Halogenated organics and PFAS

All though, Nonylphenol, Bisphenol A and DEHP were all detected in inlet, only Nonylphenol was detected in the buffer tank.

The halogenated organics were not detected in inlet or buffer at all. Therefore, surprising to see them in outlet of Line 1 in one out of four samplings.

Several PFAS connections were detected in the inlet and buffer tank.

TABLE 18. Halogenated organics and PFAS results.

Compound	Concentration	L1 (15/03/22)	L2 (15/03/22)	Buffertank (8,15,22/03/22)	Method
Nonylphenol	µg L^{-1}	<0.5	3.85	1.34-2.61	DTU-lab
Bisphenol A	µg L^{-1}	<0.5	<0.5	<0.5	DTU-lab
DEHP	µg L^{-1}	<0.5	<0.5	<0.5	DTU-lab
Tetrachlorethylene	µg L^{-1}	119.3	<10	<10	GCMS
Trichlorethylene	µg L^{-1}	87.6	<10	<10	GCMS
Chloroform	µg L^{-1}	<2.5	<2.5	<2.5	GCMS
1,1,1-Trichlorethane	µg L^{-1}	23.1	<10	<10	GCMS
PFPeA	ng L^{-1}	1.65	<1	<1	LCMSMS
PFBS	ng L^{-1}	<2	<2	<2	LCMSMS
PFHxA	ng L^{-1}	<1	<1	<1	LCMSMS
PFHpA	ng L^{-1}	<1	<1	1.32-1.83	LCMSMS
PFHxS	ng L^{-1}	<1	<1	40.67-41.60	LCMSMS
PFOA	ng L^{-1}	<0.5	<0.5	2.91-3.08	LCMSMS
PFNA	ng L^{-1}	<1	<1	<1	LCMSMS
PFOS	ng L^{-1}	<0.5	<0.5	2.50-3.36	LCMSMS
PFDA	ng L^{-1}	<1	<1	<1	LCMSMS
PFUdA	ng L^{-1}	<1	<1	<1	LCMSMS
PFOSA	ng L^{-1}	<1	<1	<1	LCMSMS
PFDoA	ng L^{-1}	<1	<1	<1	LCMSMS
PFTrDA	ng L^{-1}	<1	<1	<1	LCMSMS
PFTeDA	ng L^{-1}	<1	<1	<1	LCMSMS

8.2.3 Pharmaceuticals

Overall rejections are very consistent for all substances with either rejection to below limit of detection or close to limit of detection. A few compounds were detectable at the outlet. Except for 1H-Benzotriazol all were well below PNEC values:

Compound	PNEC	L1/L2 measured
1H-Benzotriazol	900 ng L-1	(25,000/23,000)
Ciprofloxacin	89 ng L-1	(54/23)
Diclofenac	40 ng L-1	(10/10)
Paracetamol	9,200 ng L-1	(63/ND)
10,11-dihydroxycarbamazepin	500 ng L-1	(140/280)
Acesulfame:	NA	(46/34)
Iohexol	1,000,000 ng L-1	(340/230)
Iomeprol	1,000,000 ng L-1	(1000/620)

TABLE 19. Pharmaceuticals results and overall rejections between buffer tank and outlets.

Compound	LOQ (ng L^{-1})	Method	Concentration (ng L^{-1})			Rejection (%)	
			Outlet L1	Outlet L2	Buffer tank	Outlet L1	Outlet L2
10,11-Dihydroxy-10,11-dihydrocarbamazepin	30	DIN EN ISO 21676	< 30	< 30	3,600	Below detection limit	Below detection limit
1H-Benzotriazol	30	DIN EN ISO 21676	25,000	23,000	370,000	93.2%	93.8%
4N-Acetylsulfadiazin	30	DIN EN ISO 21676	< 30	< 30	150	Below detection limit	Below detection limit
4N-Acetylsulfamethoxazol	30	DIN EN ISO 21676	< 30	< 30	4,200	Below detection limit	Below detection limit
Bezafibrate	30	DIN EN ISO 21676	< 30	< 30	420	Below detection limit	Below detection limit
Bisoprolol	30	DIN EN ISO 21676	< 30	< 30	200	Below detection limit	Below detection limit
Candesartan	30	DIN EN ISO 21676	< 30	< 30	2,100	Below detection limit	Below detection limit
Carbamazepine	30	DIN EN ISO 21676	< 30	< 30	1,900	Below detection limit	Below detection limit
Ciprofloxacin	10	DIN EN ISO 21676	54	23	34,000	99.99%	99.99%
Clarithromycin	30	DIN EN ISO 21676	< 30	< 30	77	Below detection limit	Below detection limit
Diclofenac	10	DIN EN ISO 21676	10	10	3,200	99.99%	99.99%
Diuron	30	DIN EN ISO 21676	< 30	< 30	34	Below detection limit	Below detection limit
Flufenacet	10	DIN EN ISO 21676	< 10	< 10	< 10	Not detected in influent	Not detected in influent
Gabapentin	30	DIN EN ISO 21676	< 30	< 30	180,000	Below detection limit	Below detection limit

Ibuprofen	10	DIN EN ISO 21676	< 10[a]	< 10[a]	78,000	Below detection limit	Below detection limit
Isoproturon	30	DIN EN ISO 21676	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Losartan	30	DIN EN ISO 21676	< 30	< 30	30,000	Below detection limit	Below detection limit
Sum of 4+5-Methylbenzotriazol	30	DIN EN ISO 21676	< 30[a]	< 30[a]	2,000	Below detection limit	Below detection limit
Metoprolol	30	DIN EN ISO 21676	< 30	< 30	7,300	Below detection limit	Below detection limit
Propiconazole	30	DIN EN ISO 21676	< 30	< 30	250	Below detection limit	Below detection limit
Sotalol	30	DIN EN ISO 21676	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Sulfamethoxazole	30	DIN EN ISO 21676	< 30	< 30	13,000	Below detection limit	Below detection limit
Tebuconazole	30	DIN EN ISO 21676	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Terbutryn	20	DIN EN ISO 21676	< 20	< 20	< 20	Not detected in in-fluent	Not detected in in-fluent
Valsartan	30	DIN EN ISO 21676	< 30	< 30	11,000	Below detection limit	Below detection limit
Amisulprid	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Atorvastatin	30	LC-MS/MS	< 30	< 30	13,000	Below detection limit	Below detection limit
Azithromycin	30	LC-MS/MS	< 30	< 30	2,900	Below detection limit	Below detection limit
Capecitabin	30	LC-MS/MS	< 30	< 30	440	Below detection limit	Below detection limit
Cefalexin	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Citalopram	30	LC-MS/MS	< 30	< 30	790	Below detection limit	Below detection limit
Dimethyl Benzotriazole	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Ifosfamid	30	LC-MS/MS	< 30	< 30	6,200	Below detection limit	Below detection limit
Mefenamic acid	30	LC-MS/MS	< 30	< 30	260	Below detection limit	Below detection limit
Metronidazole	30	LC-MS/MS	< 30	< 30	130	Below detection limit	Below detection limit
Miconazole	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Mycophenolic acid	30	LC-MS/MS	< 30	< 30	20,000	Below detection limit	Below detection limit
Naproxen	30	LC-MS/MS	< 30	< 30	8,900	Below detection limit	Below detection limit
Ofloxacin	30	LC-MS/MS	< 30	< 30	1,700	Below detection limit	Below detection limit

Paracetamol	30	LC-MS/MS	64	< 30	53,000	99.9%	Below detection limit
Phenazone	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Prednisolone	100	LC-MS/MS	< 100	< 100	< 100	Not detected in in-fluent	Not detected in in-fluent
Propranolol	30	LC-MS/MS	< 30	< 30	2,100	Below detection limit	Below detection limit
Roxithromycin	30	LC-MS/MS	< 30	< 30	460	Below detection limit	Below detection limit
Sulfadiazine	30	LC-MS/MS	< 30	< 30	470	Below detection limit	Below detection limit
Sulfathiazole	30	LC-MS/MS	< 30	< 30	7,700	Below detection limit	Below detection limit
Sulphapyridine	30	LC-MS/MS	< 30	< 30	4,800	Below detection limit	Below detection limit
Tramadol	30	LC-MS/MS	< 30	< 30	7,500	Below detection limit	Below detection limit
Trimethoprim	30	LC-MS/MS	< 30	< 30	220	Below detection limit	Below detection limit
Venlafaxine	30	LC-MS/MS	< 30	< 30	3,600	Below detection limit	Below detection limit
10,11-Dihydroxycar-bamazepin	30	LC-MS/MS	140	280	< 30	-	-
Allopurinol	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Amoxicillin	50	LC-MS/MS	< 50	< 50	< 50	Not detected in in-fluent	Not detected in in-fluent
Atenolol	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Azathioprine	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Bicalutamide	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Carvedilol	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Desvenlafaxine	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Glucosamine	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Guanylurea	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Imidacloprid	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Irbesartan	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Ketoprofen	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Melamine	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent

						Not detected in in-fluent	Not detected in in-fluent
Metformin	30	LC-MS/MS	< 200[b]	< 50[b]	< 30	Not detected in in-fluent	Not detected in in-fluent
Methotrexate	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Penicillin G	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Rosuvastatin	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Sertraline	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Spironolactone	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Sulfasalazine	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Zopiclone	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
4N-Acetylsulfamerazin	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
4N-Acetylsulfametha-zin	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Amiloride	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Cefazolin	-	LC-MS/MS	< 200	< 200	< 200	Not detected in in-fluent	Not detected in in-fluent
Cefotaxime	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Climbazole	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Clindamycin	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Cyclophosphamide	-	LC-MS/MS	< 30	< 30	430	Below detection limit	Below detection limit
Enalapril	-	LC-MS/MS	< 30	< 30	2,900	Below detection limit	Below detection limit
Hydrocortisone	-	LC-MS/MS	< 90	< 90	< 90	Not detected in in-fluent	Not detected in in-fluent
Metconazole	-	LC-MS/MS	< 50	< 50	< 50	Not detected in in-fluent	Not detected in in-fluent
Norfloxacin	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Propyphenazone	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Quinoxyfen	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Ranitidine	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Ritalinic acid	-	LC-MS/MS	< 30	< 30	5,300	Below detection limit	Below detection limit
Simvastatin	-	LC-MS/MS	< 9,000	< 9,000	< 9,000	Not detected in in-fluent	Not detected in in-fluent

Sulfamethazine	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Sulfamerazine	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Sulfamethazine	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Tamoxifen	-	LC-MS/MS	< 500	< 500	< 500	Not detected in in-fluent	Not detected in in-fluent
Warfarin	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Acesulfame	10	LC-MS/MS	46	34	250,000	99.98%	99.99%
Cefuroxime	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Clofibric acid	300	LC-MS/MS	< 300	< 300	< 300	Not detected in in-fluent	Not detected in in-fluent
Furosemide	50	LC-MS/MS	< 50	< 50	52,000	Below detection limit	Below detection limit
Gemfibrozil	50	LC-MS/MS	< 50	< 50	8,000	Below detection limit	Below detection limit
Hydrochlorothiazide	30	LC-MS/MS	< 30	< 30	840	Below detection limit	Below detection limit
Mecoprop	30	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Triclosan	-	LC-MS/MS	< 30	< 30	< 30	Not detected in in-fluent	Not detected in in-fluent
Amidotrizoic acid	50	DIN EN ISO 21676	< 50	< 50	91,000	Below detection limit	Below detection limit
Iohexol	50	DIN EN ISO 21676	340	230	5,300,000	99.99%	99.99%
Iomeprol	50	DIN EN ISO 21676	1,000	620	14,000,000	99.99%	99.99%
Iopamidol	50	DIN EN ISO 21676	< 50	< 50	< 50	Not detected in in-fluent	Not detected in in-fluent
Iopromide	50	DIN EN ISO 21676	< 50	< 50	18,000	Below detection limit	Below detection limit
Ioversol	50	DIN EN ISO 21676	< 50	< 50	58,000	Below detection limit	Below detection limit
Salicylic acid	100	LC-MS/MS	< 100	< 100	1,7960	Below detection limit	Below detection limit
Cimetidine	10	LC-MS/MS	< 10	< 10	< 10	Not detected in in-fluent	Not detected in in-fluent
Erythromycin	100	LC-MS/MS	< 100	< 100	291	Below detection limit	Below detection limit

8.2.4 Learnings and recommendations

This consistency over many substances indicates, that this will also be the case for other micropollutants not tested here. Given the issues with the RO membrane integrity during sampling, it indicates that the FO membrane is the main point of rejection for organic micropollutants.

Based on the results achieved, and when the final system is optimized in terms of design, materials, and operation, we believe that the system will be able to deliver a very high outlet water quality - at drinking water level.

The expected outlet quality is summarized and commented as follows:

TABLE 20. Expected water quality from the system.

Wastewater parameters			Drinking water requirements in DK
Total-COD	mg L^{-1}	<15	
Total-N	mg L^{-1}	<6	
NH$_4$-N	mg L^{-1}	<5	<0.05 mg NH4+ L^{-1}
NO$_3$-N	mg L^{-1}	<0.5	<50 mg NO3- L^{-1}
NO$_2$-N	mg L^{-1}	<0.02	<0.1 mg NO2- L^{-1}
Total-P	mg L^{-1}	<0.1	<0.15

General parameters			
Colour and Smell		None	None
H$_2$S	mg L^{-1}	<0.05 < 0.05	
pH	pH	7 – 8,5	7-8.5
Oxygen content	mg L^{-1}	>5	>5
Conductivity	mS/m	>30	<2500
Hardness, total	°dH	*	Non-aggressive

Microbiological parameters**			
Coliform bacteria	MPN/100 ml	N.D.	N.D.
Escherichia coli	MPN/100 ml	N.D.	N.D.
Total plate count at 22°C	CFU/ml	< 200	<200

Dissolved substances and metals			
Chloride	mg/l	*	<250 mg L^{-1}
Sulphate, filtered	mg/l	<3	<250 mg L^{-1}
Aggressive carbondioxide	mg/l	<15	<2 mg L^{-1}
NVOC	mg/l	<0.4	<4
Calcium (Ca)	mg/l	<2.5	
Iron (Fe)	mg/l	< 0.01	<0.2
Potassium (K)	mg/l	<10	
Mercury (Hg)	µg/l	< 0.05	<1
Magnesium (Mg)	mg/l	*	
Manganese (Mn)	mg/l	< 0.002	<0.05
Sodium (Na)	mg/l	*	<175
Silver (Ag)	µg/l	< 0.1	<10

Other substances
Pharmaceuticals and other environmental hazardous substances in general are expected to be below or close to limit of detection.

*The hardness of the water as well as concentrations for sodium and magnesium will depend on the draw salt chosen and the up-concentration level in the septic tank:

Using NaCl the hardness will be close to zero, concentrations of sodium are expected to range from 50-300 mg L^{-1} and chloride from 75 – 450 mg L^{-1}. Magnesium zero.
Using MgCl2 the hardness will depend on Magnesium concentrations, which are expected to range from 25 – 200 mg L^{-1} and chloride from 75 - 600 mg L^{-1}. Sodium zero.
(99,4% RO rejection rate, 100 L $Hour^{-1}$ draw recirculation and 50% RO recovery rate used for estimations).

**The system will be an effective double barrier against harmful human bacteria and viruses. Only nature-based bacteria may grow after the system. This may impact total plate counts over time and require disinfection of permeate and re-use system to keep values below limit at all times.

The water quality will differ from Danish drinking water requirements on a few parameters:
-    Ammonium above drinking water limits.
-    The water will be aggressive, due to the lack of calcium and too much carbonic acid.
-    Chloride that will range from below to above drinking water limits
-    Sodium that will range from below to above drinking water limits, if used as draw salt

The water quality will allow for infiltration, without risk of groundwater contamination. Ammonium is already naturally present in the ground due to degradation of organic matter and is be removed by aeration, when extracted and supplied for drinking water. Chloride and Sodium are naturally present in groundwater at different levels. Despite occasionally higher concentrations at maximum water recovery levels, the average concentrations of chloride and sodium will not pose a threat to the groundwater.

Re-use of the water is only intended for the following non-potable purposes:
-    toilet flushing
-    garden irrigation/car wash/surface wash of walking areas and roads,
-    washing machine and dishwashers.
Re-use is only intended by installation of a new separate pipe system, in materials fit-for-purpose between the system and the intended point-of-uses. The pipe system must be separated to avoid any mixing with the drinking water distribution system, and to respect all relevant legislation on this matter to avoid drinking water contamination.

The aggressive water combined with the chloride concentrations increases the corrosion potential of the water. Correct selection of materials for the new re-use water pipe system can solve this corrosion challenge and today's machines for dishwashing and laundry washing are already designed for working with similar water qualities, so the corrosion risk here is assumed low.

The chloride content will increase from start to end of a batch (75 to 450/600 mg L^{-1}) and if used for irrigation, the average will be on level with drinking water requirements (250 mg L^{-1}), therefore the risk of using the water for garden irrigation will be low.

Overall, the water quality is suitable for the intended non-potable purposes.

In addition, the water will bring some extra benefits in daily use:

- Less detergents needed due to low level of carbonates.
- Lack of calcium will reduce bacteria growth/smell issues in laundry washing machines.
- The aggressive water will remove calcium-based scaling when used for toilet flushing.

9. Power consumption

9.1 Power consumption

The power consumption has not been measured on the test plant. The test plant and state of the development process is as such too far away from being comparable to the final system for this to make sense. Instead, learnings have been made about, what must be the focus points to address, when designing the final system.

With reference to FIGURE 33 these learnings will be listed below:

a) Feed pump:

To optimize power consumption for the feed pump (marked a), the needed flow and pump head must first be minimized. This is mainly determined by the feed side of the FO membrane.

There will be a lower limit, set by the needed flux through the membrane times a factor of at least 2, to avoid too high solids concentrations that causes clogging inside the FO membrane. The expected level will be around 100 L/hour.

The type of pump must be carefully selected to address the given operating conditions (particle sizes, fluid characteristics, flow, and pump head) to achieve the highest possible pump efficiency.

b) FO membrane

The layout of the system, and with that the height and diameter of the FO membrane, will be important, to minimize the static head loss in the system. Though, the biggest head loss will be inside the FO hollow fibers, and therefore this calls for a FO membrane with higher hollow fibers ID, shorter length and with that also a larger external module diameter. The hollow fibers ID needs to be balanced, as there are two drawbacks with an increased ID. The higher ID the thicker the supporting fiber needs to be. This increases the osmotic pressure difference needed to operate the membrane, which again increases power consumption. Furthermore, it also increases the cost of the membrane.

In addition to this, the FO membrane cake layer control mechanism must be simplified, so it works with no excessive flows or pressure, as due to cost issues, the mechanism employed can only involve the feed pump already in place. There will not be economy for a second and larger pump.

c) High-pressure pump

The high-pressure pump (marked b) is used to feed the RO membrane with sufficiently large pressure to get the required permeate flow. This is the highest power consuming element in the entire system, and therefore it needs to be optimized carefully. The first way is to use a pressure energy recovery device (marked c) on the brine outlet from the RO membrane, as this will cut up to 80% of the power consumption. Furthermore, it is important first to focus on the recovery rate (permeate flow / feed flow) of the RO membrane, as this will reduce the needed draw circuit recirculation flow. A recovery rate up to 40-50% should be the aim, leaving the required recirculation flow around the 100 L/hour level. This will impact the layout of the RO membrane, that needs to be optimized in terms of length and diameter ratio, so the salt concentration inside the RO does not start to cause precipitations/clogging.

d) Motor for pumps

This is a relatively small system; therefore, the motor efficiency is of relatively high importance due to friction-losses. Motors will be needed for the feed pump and the high-pressure pump. Though, instead of two smaller one-axle motors it makes sense to investigate one larger double-axle motor, as motor efficiency increases with larger motor sizes.

In addition to this, motors should be permanent magnet types and of course size matched perfectly with the load to maximize the efficiency.

In addition to the abovementioned mechanical design issues, the power consumption will be low in the beginning of a cycle, as the osmotic pressures are low, and highest at the end of cycle, where the osmotic pressures are high. The realized average kWh/m3 power consumption will therefore depend on the amount of m^3 treated. For an average Danish family with 2,15 persons this is around 80 m^3 per annum, whereas summer houses are around 30 m^3 per annum. At present it is therefore not possible to come up with a better and more precise power consumption of the final system than already stated.
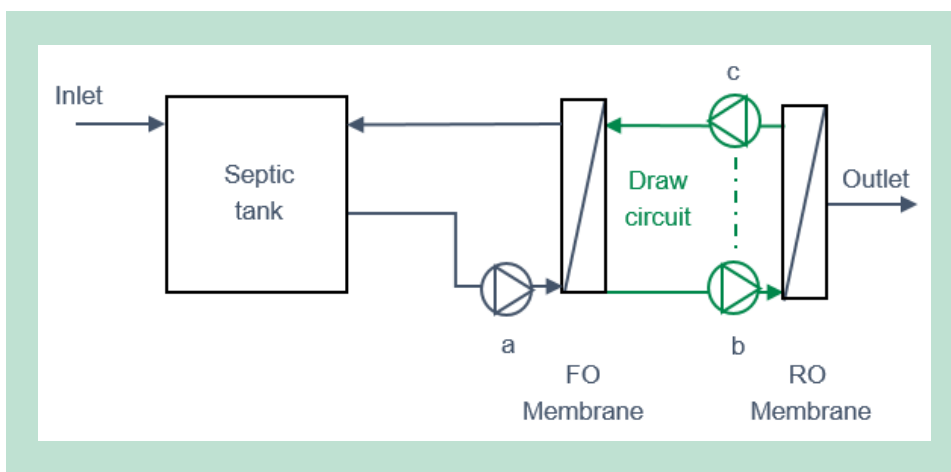

FIGURE 33. Plant diagram with main components.

10. Concentrate stability and biogas potential

The biochemical methane potential (BMP) was preliminarily assessed for the up-concentrated feed (the 1% left after having extracted 99% of the water) after the initial lab test.

Main concerns could relate to inhibition due to the i) high salinity resulting from both up-concentration and reverse salt flux; ii) organic overload due too high concentration of highly biodegradable organics or organic acids and iii) high ammonia. Accumulation of heavy metals and organic micropollutants are also of concern, as they may limit the valorisation pathways of the digestate.

The preliminary essay shows a lag phase compared to the control (see FIGURE 34), but after this is overcome, the concentrate shows a high BMP.

The methane production potential (B_∞) and the hydrolysis constant (k_h) were estimated using MS Excel (2019) and first-order kinetics:

$$B_t = B_\infty(1 - e^{-k_h \cdot t})$$

where B_t (mL CH$_4$ g VS^{-1}) is the cumulative methane produced until a given time t. The average methane yield was calculated at 1 atm and 0°C, based on the triplicate batch test conducted as basis for the experiment.

The biomethane production potential (B_∞) was estimated to 648 mL CH$_4$ g VS^{-1}, which is significantly higher than levels commonly reported for secondary sludge from municipal wastewater treatment plants (around 200 mL CH$_4$ g VS^{-1}) [7].
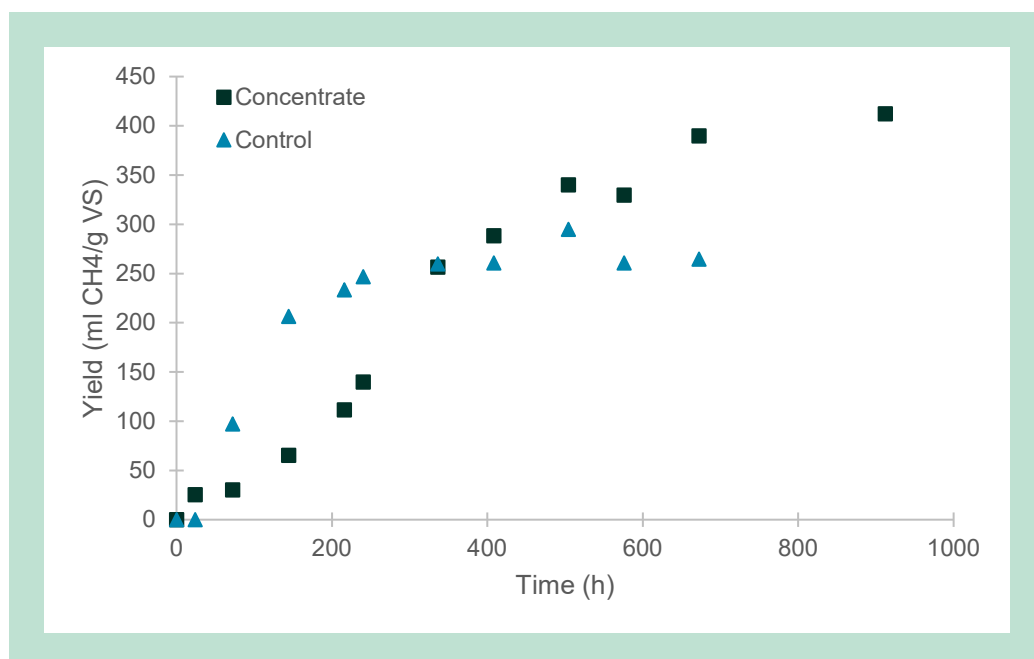

FIGURE 34. Methane production per gram of added volatile solids (VS) as a function of time

Throughout the test, concentrations of methane (CH_4) and carbon dioxide (CO_2) have been sampled from the feed line after the bottom of the buffer tank. Results can be seen in TABLE 21.

The results document that there is almost no methane production in the septic tank. The low levels of carbon dioxide document that there is also limited oxidation of organic matter taking place.

Organic acids were monitored in the bottom sediments of the septic tank, where most microbial biomass stayed at anaerobic conditions, which poses risk of fermentation. However, organic acids were low in concentration, with the presence of acetic acid (<5 mg/L) and iso valeric acid (<2.9 mg/L). Therefore, the sediments had experienced little microbial activity, which is in line with the low methane levels detected.

The low level of oxygen combined with salinity levels might explain the overall low anaerobic and aerobic biological activity and with that little loss of biomethane potential over time.

As documented above in section 8 the levels of Nitrate and Nitrite are overall very low too (except from a period with very high recirculation/distribution of oxygen in the septic tank), which documents that a low recirculation rate will lead to a low level of oxygen, that will result in a low biological oxidation of ammonium into Nitrite and Nitrate. With a low transformation of ammonium, a negligible emission of nitrous oxide (N_2O) is expected too.

Overall, this indicates that the emission of the harmful greenhouse gasses methane and nitrous oxide will be very low for the system. In addition, organics are not degraded, but preserved with a higher biomethane potential of the concentrate as a result.

TABLE 21. Methane and carbon dioxide concentrations in feed. Buffers 1 and 2 are duplicates of the same grab sample. Throughout the test, the pH has ranged between 7.7 and 8.7 with an average around 8.2.

Date	Sample ID	CO_2	CH_4	CO_2 in water	CH_4 in water	CH_4 in air at 20°C
		%	%	mg/l	mg/l	mg/l
01-03-2022	Buffer_1	0,93	0,21	27	0	2
	Buffer_2	0,92	0,08	27	0	1
08-03-2022	Buffer_1	1,01	0,00	30	0	0
	Buffer_2	1,17	0,00	34	0	0
29-03-2022	Buffer_1	1,77	0,08	52	0	1
	Buffer_2	1,50	0,00	44	0	0
12-05-2022	Buffer_1		0,42		0	4
	Buffer_2		0,35		0	4
20-05-2022	Buffer_1		0,50		0	5
	Buffer_2		0,00		0	0
05-07-2022	Buffer_1	0,53	0,00	15	0	0
12-07-2022	Buffer_1	0,28	0,00	8	0	0
19-07-2022	Buffer_1	0,76	1,44	22	1	15
06-09-2022	Buffer_1	0,42	0,00	12	0	0
22-09-2022	Buffer_1	0,26	0,00	8	0	0

At the end of the project run, a biochemical methane potential (BMP) test was repeated on the septic tank content (mid and bottom phases tested and averaged). As can be seen in FIGURE 35 the inoculum used was unable to transform the septic tank content into biomethane. The control also demonstrates lower activity of the inoculum compared to the first experiment.
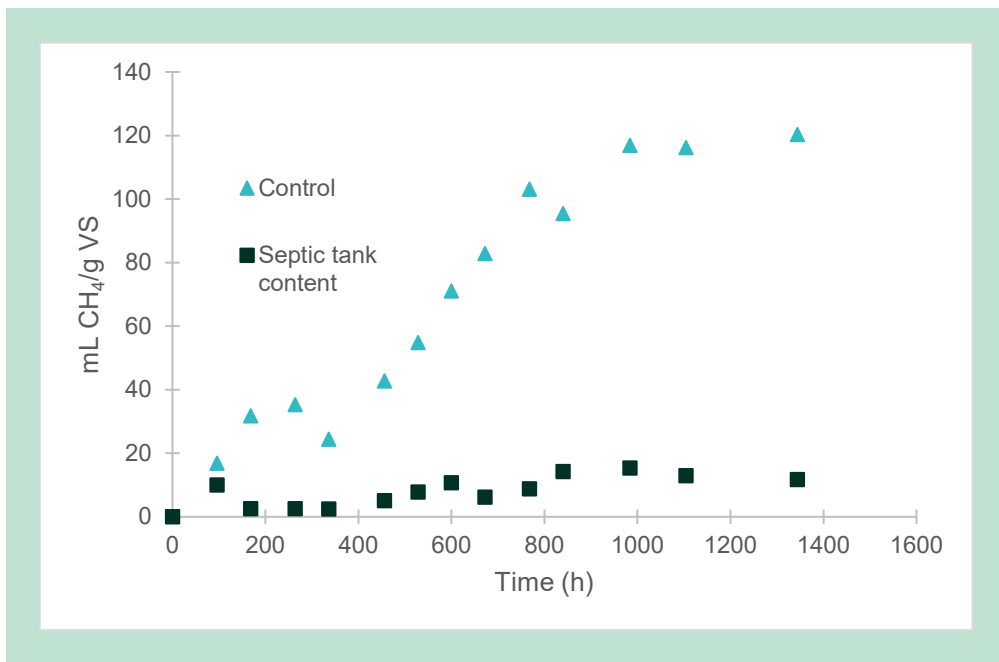
FIGURE 35. Methane production per gram of added volatile solids (VS) as a function of time.

The explanation why the inoculum could not turn the septic tank content into biomethane is most likely a combination of the test inoculum not being adapted to the salinity level of the septic tank content and that the overall activity level of the test inoculum was lower than in the first experiment.

Anaerobic digesters can adapt to the salinity levels experienced in our concentrates within weeks, where constant feeding of the higher salinity substrate will slowly select for those microbes more tolerant to salt stress, keeping high methane yields. In full scale applications the septic sludge may also be mixed and co-digested with conventional secondary sludge making this adaptation go faster.

11. References

[1]   DHI, »Full scale advanced wastewater treatment at Herlev Hospital,« DHI, May
        2016.

[2]   M. K. Jørgensen, »Effect of reverse sodium flux and pH on ammoniacal
        nitrogen transport,« Separation and Purification Technology 217, pp. 40-47, 1
        February 2019.

[3]   J. M. G. a. A. K. Suresh, »Clorine Attack on reverse osmosis membranes:
        Mechanisms and mitigation strategies,« Journal of Membrane Science,
        Volume 541, pp. 108-126, 1 November 2017.

[4]   The Danish Environmental Protection Agency, *Miljøstyrelsens Vejledning for
        nedsivningsanlæg op til 30 PE -,* Miljøstyrelsen, 2000.

[5]   L. W. J. J. L. H. T. J. Mogens Henze, Spildevandsteknik, Polyteknisk Forlag,
        2009.

[6]   M. W. A. P. a. A. S. D. Breite, »The critical zeta potential of polymer
        membranes: how electrolytes impact membrane fouling,« *Royal Society of
        Chemistry Advances,* pp. 98180-98189, 9 October 2016.

[7]   D. K. I. A. Mariusz Kuglarz, »Microwave and thermal pretreatment as methods
        for increasing the biogas potential of secondary sludge from municipal
        wastewater treatment plants,« Bioresource Technology, Volume 134, pp. 290-
        297, 2013.

Appendix 1. A Head loss calculation

HEAD LOSS CALCULATION FOR 0.8 MM ID FO MODULE

Input	Unit	Number	Number	Number
HF internal diameter ID	mm	0.8	0.8	0.8
FO module membrane area	m2	6.8	6.8	6.8
Total length of HF	m	2706	2706	2706
FO module length	m	1.7	1.7	1.7
Total number of HF	stk.	1592	1592	701
Blocked number of HF	%	0%	0%	56%
Cake layer reduction of ID	mm	0	0.15	0
Effective HF cross area	m2	0.00080	0.00053	0.00035
Diameter before/after HF	mm	95	95	95
Cross area before/after HF	m2	0.00709	0.00709	0.00709
FO inflow	L/time	392	392	392
FO flux in operation	LMH	2	2	2
FO flux	L/time	13.6	13.6	13.6
FO outflow	L/time	378	378	378
Velocity before HF	m/s	0.015	0.015	0.015
HF inflow velocity	m/s	0.136	0.205	0.309
HF outflow velocity	m/s	0.131	0.198	0.298
Velocity after HF	m/s	0.015	0.015	0.015
Density and kinematic viscosity as		vand	vand	vand
Liquid temperature	°C	30	30	30
Density	kg/m3	996	996	996
Kinematic viscosity	m2/s*1E6	0.801	0.801	0.801
Head loss calculations				
Effective HF ID	m	0.0008	0.000652	0.0008
Reynolds number <2000	-	136	167	309
Frictions coefficient. Laminar.	-	0.47	0.38	0.21
HF length	m	1.7	1.7	1.7
Head loss in HF	Pa	9227	20945	20955
Area ratio HF/before HF	-	0.11	0.07	0.05
Contraction coefficient	-	0.62	0.61	0.61
Head loss coefficient	-	0.38	0.40	0.41
Head loss FO inlet	Pa	3	8	20
Area ratio HF/after HF	-	0.11	0.07	0.05
Head loss coefficient	-	0.79	0.86	0.90
Head loss FO outlet	Pa	7	7	8
Total head loss FO module	**Pa**	9238	20961	20982

Total head loss FO module	**mVS**	0.94	2.14	2.14
Total head loss FO module	**bar**	0.09	0.21	0.21
Static pressure at sensor	**bar**	0.09	0.09	0.09
Total pressure at sensor	**bar**	0.18	0.30	0.30

Appendix 2. Logbook

Date	Events
Start-up 1	
07/10/2021	• FO modules 0.2mm ID type HFFO14 #172 on Line 1 and #146 on Line 2 • Filter1 - Fibertex F-20 • Pilot put into operation with wastewater • Filter1 - shows immediate challenges with lack of water flow at 5 cm driving water level • Filter1- Driving water level increased to 15 cm in buffer tank and FO feed flow reduced to app. 180 L/hour/line. • P1 Piston pumps show large fluctuations in FO inlet pressure from 0.1 – 1 bar(g)., • P2 high-pressure pump limited to 80% capacity due to lack of feed pressure.
07/10/2021 – 10/10/2021	• Intermittent operation with multiple stops due to lack of water flow across Filter1.
10/10/2021	• Filter1 replaced with Fibertex F-10 • improved water flow across pre-filter
10/10/2021 – 19/10/2021	• Continuous operation
19/10/2021	• FO hollow fiber rupture – Brown water in draw tanks. • Feed osmotic pressure 0.063 Osm/kg, as expected relative to amount of water treated • **Water treated: 5 m^3** (2,362 L on Line 1 and 2,722 L on Line 2)
19/10/2021 – 25/10/2021	**Standstill**
Start-up 2	
25/10/2021	• Installation of new FO modules 0.2 mm ID type HFFO14 #466 on Line 1 and #467 on Line 2 • New draw solutions on both lines. • Pilot re-start
25/10/2021 – 28/10/2021	• Irregular operation due to emergency stops related to insufficient flow across Filter1
28/10/2021	• Filter1 replaced with SEFAR 0.15 • Filter1 also shows low water flow • H$_2$S formation observed in draw tanks and in permeates
29/10/2021	• Feed osmolarity at 0.1 osmol/kg which is higher than expected, indicating salt loss from draw to feed or from stop draw tanks. • Difference between draw osmotic pressure (21 bar) and RO pressure required for permeate production (49) is 28 bar, indicating severe RO fouling • Osmotic pressure difference over the FO membrane around 18 bar. • Permeate production around 50 L/H – (3.6 LMH).
29/10/2021 – 01/11/2021	• Irregular operation • **Water treated: 5.5 m^3**
02/11/2021	• **Standstill** • Stop due to FO breakdown – brown water in draw tanks.
08/11/2021	• FO membrane inspection shows clogging at the inlet to the FO membranes indicating that pre-treatment is not adequate.
18/11/2021	• RO membrane cleaning with alkaline and acid.
02/12/2021	• P1. Piston pump replacement to impeller type pump that secure much more constant pressure and flow conditions on FO feed. • Additional Filter2 installed to protect FO membrane

	• New pump P3 installed in draw circuit to ensure sufficient feed pressure and 100% flow capacity on P2 high-pressure pumps
Start-up 3	
03/12/2021	• Filter1 replaced with SEFAR 0.2 mm • Filter2 installed with 50 μm in-depth cartridge filter type polypropylene wire. The filter and housings do not seal tightly around the ends, so filter may not work 100% as intended. • Pilot re-start
03/12/2021 – 05/12/2021	• Continuous operation. • FO flux and RO pressure levels around < 2 LMH and 16 bar(g) on both lines. • Stop due to low level in draw tanks / lack of salt.
06/12/2021	• Bucket tests shows P1 does not deliver the expected flow on both lines and very sensitive to back pressure – so actual flow during operation is assumed close to zero (later discovered that this was due to wrong motor wiring). • Filter2 replaced daily. • Osmotic pressure difference between the FO feed (0.14 Osmol/kg) and the draw tanks (0.576 – 0.714 Osmol/kg) was >10 bar, with a flux (< 2 LMH). • Osmolarity in the feed is rising faster than expected and is assumed related to salt loss, earlier FO membrane failure and salt from stop processes. • Sampling for particle size analysis for inlet, septic tank, and after Filter1
06/12/2021 – 13/12/2021	• Intermittent operation • Daily stops due to low level in draw tanks. • **Water treated: 6.2 m^3**
13/12/2021	• FO module assumed clogged.
17/12/2021 – 20/12/2021	• FO module cleaning at AQP with hot water, osmotic backwash, alkaline cleaning, and acid cleaning • Change of biocide loop. Return flow to biocide canisters removed as this consumes the disinfection capacity
Start-up 4	
20/12/2021	• FO module (HFFO14 #466 and #467) re-installed. • New draw solution • Filter1 (SEFAR 0.2 mm) shows no signs of blocking • Filter2 (50 μm) cartridge filters replaced with new 50 μm in-depth filters type blow moulded polypropylene.. • P1 feed pumps deliver around 210 L/h (bucket test) at start (pump issues still prevailing). • Pilot re-start
20/12/2021 – 29/12/2021	• Plant operation start with RO operating pressure set to just below 20 L/h (40 bar on Line 1 and 20 on Line 2), indicating membrane fouling issues. • Continuous operation, stop 22/12 due to power outfall. • **Water treated: 6.3 m^3**
29/12/2021- 05/01/2022	• **Standstill** • FO module breakdown. brownish waters present in the draw tanks. Plant stopped manually.
05/01/2022	• Sampling for particle size distribution analysis after Filter1 and Filter2
05/01/2021 – 17/01/2021	• Standstill and QC of new FO modules • Filter2 cartridge filter housing changed to double O-ring connection.
Start-up 5	
19/01/2022	• New FO modules installed (HFFO14 #418 on Line 1 and #439 on Line 2) • Filter2 replaced by 10 μm cartridge in-depth filter type melt blown polypropylene • P1 feed pump problems stilled prevailed. • New draw solutions.

19/01/2022 – 21/01/2022	• Irregular operation with only few hours of operation before Filter2 (10 µm) needs replacement.
21/01/2022 – 07/02/2022	• Filter2 replaced by 25 µm cartridge in-depth filter type melt blown polypropylene to increase operation time. • Intermittent operation with frequent Filter2 replacements and draw tank low level stops • P1 feed pump problems still prevail. Feed flows seen close to nil 24/1. • Suspicion of RO-leak. Permeate dripping even at high osmotic pressures in draw solution 24/1. • Suspicion of blockage in FO Line 2. Feed flow low/nil and stop due to draw tank low level. • Draw solution recirculation with "waterfall" to strip off H_2S in place. • **Water treated: 7.4 m^3**
07/02/2022 – 22/02/2022	• **Standstill** • New prototype 0.8 mm ID FO modules on both lines (M1#06 on Line 1 and M1#11 on Line 2) • LT2 level transmitter installed in pipe between draw tank and P3 circulator pump to monitor draw tank level (level reading is influenced by flow). • P1 feed pump issue solved. Discovery and correction of wiring on feed pumps, wrong wiring had resulted in lack of feed flow.
Start-up 6	
22/02/2022	• Pilot re-start • Feed flow 180 l/hour on both lines • Line 1 commissioned with NaCl and Line 2 with MgCl$_2$ • Filter2 (25 µm) filters replaced with new ones. • New RO modules on Line 2 installed. Line 1 still uses original RO modules. • Clean water is now used for refilling the stop draw tank.
22/02/2022 – 15/03/2022	• Intermittent operation • Filter2 (25 µm) needs frequent replacements • **Water treated 8.6 m^3**
15/03/2022	• Sampling for micropollutants L1/L2 and outlet water quality on Line 1
16/03/2022 – 25/03/2022	• Irregular operation • Frequent Filter2 (25 µm) exchange • Permeate productions irregular – problems with low level in draw tanks
25/03/2022	• Filter2 replaced by washable 50 µm surface filtration filters with tubular filter net in polyester.
25/03/2022 – 30/03/2022	• Continuous operation. • Filter2(50 µm) can now work for days. • Increasing problems with low level in draw tanks – no/low permeate.
30/03/2022	• O-rings replaced on RO modules on both lines to prevent leaking • New RO modules installed on Line 1 as well.
30/03/2022 – 05/04/2022	• Irregular operation due to low level in draw tanks – almost no permeate production (less than 20 L over a week for both lines) • **Water treated 8.8 m^3**
05/04/2022	• Sampling for outlet water quality in Line 2
05/04/2022 – 21/04/2022	• Continuous operation • No/low permeate production below limit of detection. • Outlet flow meters unstable since 22/2 with no readings or flows below limit of detection. • Presence of oxygen in the feed observed. • Oxygen sampling points increased to FO influent and effluent and draw influent and effluent.
21/04/2022	• Filter2 replaced by washable 70 µm surface filtration filters with filter net in stainless steel.

21/04/2022 – 06/05/2022	• Continuous operation • Since 25/4 feed flows starts a steady decline on Line 2 going to less than 50 L/hour since 2/5. RO pressure 27-30 bar until 2/5 when permeate production was stopped. • Line 1 feed flow starts a similar decline, significantly from 6/5 and going to around 100 L/hour in mid-May. • No permeate production. • Draw loop recirculation "waterfall" reverted to no "waterfall"-aeration to help determine origin of oxygen presence in feed.
06/05/2022	• Indication of blocking on FO module on Line 2. Inlet pressure has reached 2 bar(g) on Line 2 and 1 bar(g) on Line 1. Feed flow between 0-50 L/hour on Line 2 since 2/5
06/05/2022 – 17/05/2022	• Intermittent operation. • Line 1. Permeate production below limit of detection on the flow-meter. • Problems with low pressure levels and unstable flow in the draw loops on both lines starts to prevail (cause turned out to be blocked filters before high-pressure pumps), especially after 13/5. • Line 1 manometer before RO broken/leaking • Feed flow on Line 2 mostly below 50 L/hour. Permeate production stopped except from 12-14/5 with RO pressure 12 bar(g). • Absence of "waterfall"-aeration does not lead to significant production/increase of H_2S in the outlet plus oxygen concentration decreases in feed, but still present in higher concentrations than expected • **Water treated 9 m^3**
17/05/2022	• **Standstill** • Pilot operation stopped due to blocking on feed side
17/05/2022 – 07/06/2022	• Unsuccessful attempts at CIP on site
07/06/2022- 29/06/2022	• FO module cleaning at AQP. • New Biofilm sensor installed on feed Line 2. • New flow metres installed on permeate outlets with lower limit of detection • New level sensors installed directly in draw tanks. More accurate and independent on recirculation flow. • Broken manometer replaced on Line 1 (before RO) • New impeller on feed pump Line 1 – existing was broken.
Start-up 7	
29/06/2022	• Pilot re-start • Osmotic pressure in draw tanks set to 10 bar • FO feed flows set to 100% = up to app. 800 L/hour.
29/06/2022 – 10/07/2022	• Intermittent operation at around 10-15 bar RO pressure. with only occasionally low flux on both lines. • Stop due to low draw tank levels • Problems with keeping recirculation flows on both lines. • Problems with keeping feed flow on Line 1 (new impeller too soft). • **Water treated 11.8 m^3**
11/07/2022 – 24/07/2022	• Continuous operation with RO pressures up to 15-20 bars • Line 1 has stable periods with FO fluxes around 0.85 LMH at osmotic pressure differences around 17 bar. • Line 2 has stable periods with FO fluxes around 0.54 LMH at osmotic pressure differences around 14 bar. • **Water treated 14 m^3**
25/07/2022	• Pilot stopped as fouling (not complete blocking) of FO modules requires CIP at Aquaporin to prevent membrane break down. • L1 manometer failure again before RO
25/07/2022 – 09/08/2022	• Standstill and cleaning of FO membranes at AQP
09/08/2022	• Membrane failure present after cleaning (hot water, alkaline and citric acid cleanings)

	• FO from L1 (M1#06): A=0.88LMHbar, NaCl rejection: 67.04% • FO from L2 (M1#11): A=1.07LMHbar, NaCl rejection: 13.01%
10/08/2022	• Line 2 commissioned with FO membrane M1#06 and NaCl • New impeller in feed pump Line 1 (correct hardness/flow capacity) • New clean filters before high-pressure pumps on both lines (recirculation flow now stable) • Line 1 stopped (FO membrane still under cleaning)
10/08/2022 – 28/08/2022	• Continuous operation with daily stops due to heavy loss of salt/low level in draw tank Line 2 (due to membrane failure). • RO pressure around 30 bar and around 2 LMH FO flux. • 24/8- H2S present again in draw tanks and permeate. • **Water treated 19 m3**
29/08/2022	• Line 1 commissioned with FO membrane M1#11 and NaCl • FO feed pressure sensors moved so they measure FO membrane inlet pressure directly (from before pre-filter). • Upon start, more sediment was leaving the FO membrane, indicating that the cleanings had not removed all sediment. • "waterfalls" initiated again on both lines to eliminate H2S.
29/08/2022- 09/09/2022	• Continuous operation with daily stops due to heavy loss of salt/low level in draw tank Line 2 (due to membrane failure). • Feed flow setpoints 400 L/hour on both lines. • No permeate extraction on Line 1 • App. 30 bar RO pressure and 2 LMH FO flux on Line 2 • **Water treated 21.5 m³**
09/09/2022	• Permeate extraction stopped on both lines • Feed flow 200 L/hour on both lines + 30 min at 10% 2 x daily
12/09/2022	• FO Line 1 looks fine. Slight increase of FO inlet pressure over the weekend. • FO Line 2 shows signs of blocking as inlet pressure is around 0.8 bar at <200 L/hour. Manual cleanings on site. FO membrane turned, so inlet turns outlet and flushing with water for 2 hours. • Both lines restarted with feed flow 200 L/hour on both lines + 30 min stopped 2 x daily
17/09/2022	• Line 2 stopped, and FO membrane flushed with water at 600-700 L/hour. Inlet pressure reached 2.5 bar and then pump impeller broke after few hours of flushing.
17/09/2022- 18/09/2022	• Line 1 operated with 100 L/hour and 2x 30 min stop (10% flow). Stopped 18/9 due to low pressure in front of P2. • Line 2 stopped.
18/09/2022- 29/09/2022	• **Standstill** • New impeller in feed pump Line 2. • Control system changed so feed pumps 0% flow) during stop.
29/09/2022- 30/09/2022	• Both lines restarted with 100 L/hour and 2x30 min stop (0%) • Both FO inlet pressure around 0.26 bar, so Line 2 membrane recovered! • **Water treated 21.5 m³**

The non-biological on-site treatment system

The project has built up knowledge, experience and insight that makes it possible to make an on-site wastewater treatment system based on physical-mechanical functional principles, where a treatment step consisting of Forward Osmosis (FO) and Reverse Osmosis (RO) is connected directly to a standard septic tank. Clean water is drawn out of the wastewater and retained substances are concentrated in the septic tank. The project has shown that the technology can purify the water to a very high quality on level with drinking water, suitable for laundry, toilet flushing and garden irrigation purposes and which can be infiltrated into the ground without risk of groundwater contamination. The project has shown that the technology contributes to very low biological activity in the septic tank, which results in low emission of greenhouse gases and an extraordinarily high biogas potential from the sludge in the septic tank. The project has demonstrated a need for the core components of the plant to be adapted in order to realize a competitive finished product.