

Innovative pre-treatments for reverse osmosis to reclaim water from biotech and municipal wastewater for the industrial symbiosis in Kalundborg

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ABSTRACT

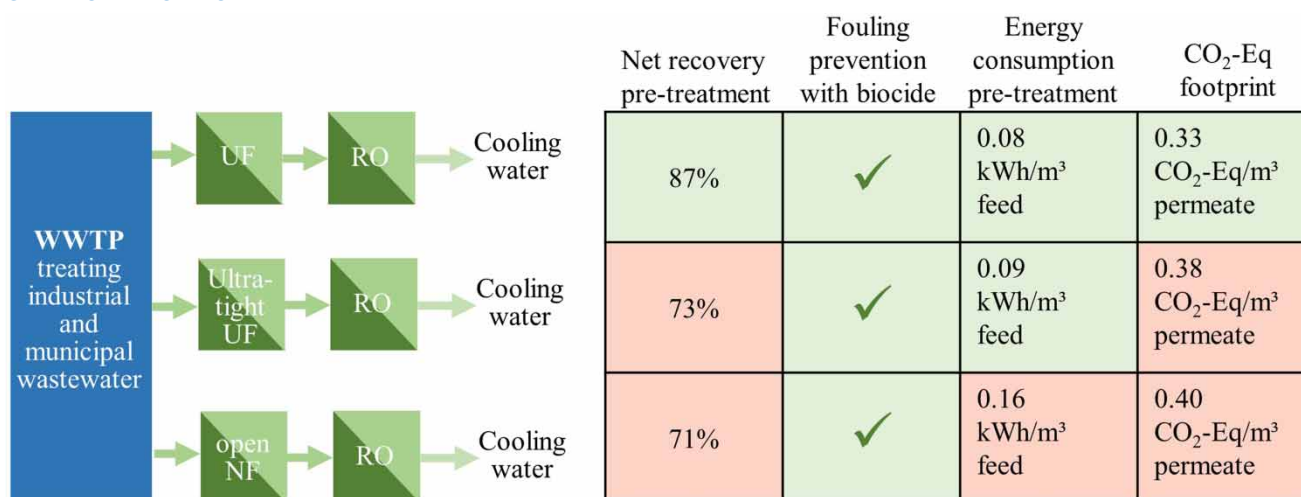
The challenge of water reclamation using membranes in this study was the quite unique wastewater composition resulting from a high share of biotech wastewater. The high content of organic matter and high concentrations of calcium, bicarbonate, and sulphate were considered as challenging for membrane processes. Consequently, an innovative ultra-tight ultrafiltration (u-t UF) membrane was developed and tested on-site at pilot scale. In comparison, a conventional UF and an open nanofiltration (NF) were piloted. The aim was to find the best pre-treatment option for reverse osmosis (RO) to reduce fouling and scaling and produce fit-for-purpose water; for example, cooling. Overall, the quality of the currently used water source was surpassed by the pilot plant. Only a standard post-treatment of the RO permeate was necessary for stabilisation. Results indicated that denser membranes only minimally reduced fouling of RO. An assessment comparing the treatment trains in a life cycle assessment using the data collected from the pilot operation (UF/NF operating settings, RO plant performance, and the design of multi-stage industrial scale RO) revealed lower greenhouse gas emissions compared to seawater desalination. However, if the RO brine treatment becomes mandatory, the greenhouse gas emissions from water reclamation and supply will be higher than those from freshwater supply.

Key words: biotech wastewater, life cycle assessment, nanofiltration, reverse osmosis, ultrafiltration, water reuse

HIGHLIGHTS

- Minor differences in reverse osmosis (RO) protection between all membranes were tested.
- Preferred option: ultrafiltration (UF) (lowest footprint and highest recovery).
- Water recovery: increased energy demand and carbon footprint when replacing freshwater from Lake Tissø, but lower when replacing seawater desalination.
- Water reclamation reduces P and N loads to Great Belt, although N concentrations in the RO brine increase compared to the secondary effluent.

GRAPHICAL ABSTRACT



ABBREVIATIONS

CEC	chemical-enhanced cleaning
CIP	cleaning in place (chemical cleaning)
COD	chemical oxygen demand
CSO	combined sewer overflows
DBNPA	2,2-dibromo-3-nitrilopropionamide (biocide)
iWW	industrial wastewater
MWCO	molecule weight cut off
mWW	municipal wastewater
NF	nanofiltration
PE	population equivalent
PES	polyethersulphone
PVP	polyvinylpyrrolidone
pWW	power plant wastewater
RO	reverse osmosis
TOC	total organic carbon
TMP	transmembrane pressure
UF	ultrafiltration
u-t UF	ultra-tight ultrafiltration
WSIS	water smart industrial symbiosis
WWTP	wastewater treatment plant

1. INTRODUCTION

As climate change intensifies, limited resources such as freshwater can seasonally become very scarce in certain regions. In 2019, 38% of Europe's population was affected by water scarcity (European Commission 2024). After agriculture, industry is the largest water-consuming sector. Water availability is a key factor for certain industries such as in the biotechnology sector, food and beverage industry, chemical and petrochemical industries. A new form of partnership between industry and the water sector has emerged as the basis for a circular economy solution: Water smart industrial symbioses (WSISs). This is where industry works with the water sector for mutual benefit. Valuable wastewater, produced by the industry, is used by the water sector to recover water, materials and/or energy, enabling their reuse by the industry again.

In Europe, internal water reuse in industry is already quite common. However, water recovery from municipal wastewater (mWW) and reuse has been implemented only in only a few countries until now and in most cases for agriculture or other irrigation purposes. Countries such as Portugal, Spain, France, Italy and Greece have already implemented their own national water reuse legislation (Ramm & Smol 2023).

Thermal (e.g. multi-effect distillation) or membrane-based (e.g. reverse osmosis (RO)) processes can be used to recover water from wastewater. Prior to desalination, a suitable and efficient pre-treatment is essential to protect the subsequent desalination process from particles and/or fouling. Fouling is the undesired formation of organic and/or inorganic deposits on membrane surfaces and is considered as a major problem in wastewater reclamation plants (Peña *et al.* 2013). Because a spiral wound RO membrane cannot be cleaned hydraulically, the cleaning process is more time consuming and has to be done manually. In contrast, a hollow fibre membrane can be cleaned by backwashing and automatic chemical cleaning procedures. Therefore, these membranes are often used to protect an RO from particles and fouling. Microfiltration (MF), ultrafiltration (UF) or nanofiltration (NF) membranes can be used as pre-treatment technologies. The combination of UF with RO is currently considered the most promising configuration due to its good intermediate retention capacity and its lower cost compared to NF (Poirier *et al.* 2023). However, the NF can achieve higher retention rates due to its smaller pore sizes compared to a UF and may better protect a subsequent RO from fouling. In combination with the membrane pre-treatment, chlorination of RO feed water is often used to further reduce biofouling in particular (Da-Silva-Correa *et al.* 2022). However, chlorine forms harmful by-products and can damage the subsequent RO membrane. Therefore, non-oxidising biocides have been developed. DBNPA is one of the most effective non-oxidising biocides providing ideal anti-biofouling properties, but is also very hazardous (Da-Silva-Correa *et al.* 2022). Therefore, alternatives are needed to further promote an environmentally friendly water reclamation and reuse.

In the European H2020 project ULTIMATE, WSIS Kalundborg is one of nine case studies. The WSIS has existed since 1972 and connects 17 companies. They already exchange various water, material and energy flows. However, water reuse is not common in Denmark and the new EU regulation on minimum requirements for water reuse has not yet been implemented until now (Ramm & Smol 2023), but water reuse has a high potential, especially in this case study. The effluent of the municipal wastewater treatment plant (mWWTP) is slightly more than 7 Mio. m³/a, which provides enough water to potentially meet the cooling needs of the WSIS. However, more than 50% of this wastewater originates from the biotech and pharmaceutical industry and has a challenging chemical composition. Therefore, it was important to first investigate in a pilot plant which treatment train is suitable for this wastewater composition. Different pre-treatment membranes such as a commercial UF, a novel ultra-tight UF and an open NF were tested to reduce the potential for biofouling on the subsequent RO. Furthermore, the potential to eliminate the need for biocide treatment of the pre-treatment effluent by using denser membranes was tested with the aim of producing cooling water quality with the proposed treatment trains. Based on the results of the pilot experiments, a full-scale basic design of the best solution was made and both, the CO₂e and the water footprints were determined.

2. METHODS

2.1. Description of the mWWTP and pilot plant

2.1.1. Municipal wastewater treatment plant

The Kalundborg mWWTP received three different wastewater streams (Figure 1): mWW (47%), pre-treated industrial wastewater (iWW; 50%) and wastewater resulting from a nearby power plant (pWW; 3%). The pre-treated iWW contained a high

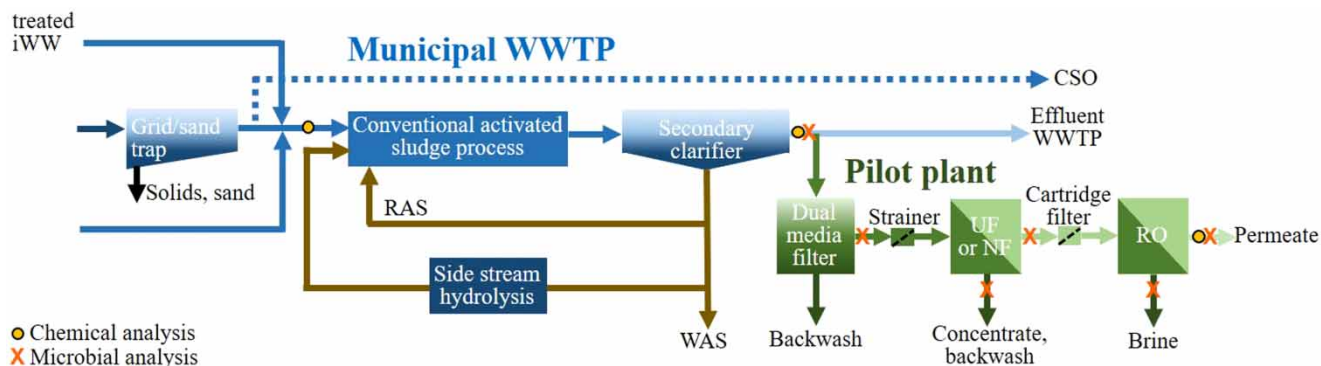


Figure 1 | Municipal wastewater treatment plant with pilot plant for water reclamation (one scheme shown for actually two treatment train operated in parallel: UF + RO, ultra-tight UF + RO or NF + RO) implemented in a side stream after the secondary clarifier. CSO, combined sewer overflow; iWW, industrial wastewater; mWW, municipal wastewater; pWW, power plant wastewater; RAS, return-activated sludge; WAS, waste-activated sludge; UF, ultrafiltration; NF, nanofiltration; RO, reverse osmosis.

fraction of non-degradable organic matter. Only the mWW was subjected to mechanical pre-treatment before all streams were mixed and transferred to the activated sludge tanks.

The mWWTP applied the conventional activated sludge process including nitrification, denitrification and enhanced biological phosphorus removal. Side stream hydrolysis was used to enhance the biological phosphorus removal. In addition, the phosphorus was removed chemically.

2.1.2. Pilot plant

The secondary clarifier effluent was used for the water reclamation pilot plant. A dual media filter was tested upstream of the two treatment trains, which were operated simultaneously. Each train consisted of either a UF, ultra-tight (u-t) UF or NF module combined with an automatically flushed pre-filter (strainer, 300 μm screen) followed by a cartridge filter and an RO module. The molecular weight cut off (MWCO) of the UF, u-t UF and NF was 150, 4 and 1 kDa, respectively. All three pre-treatment membranes were hollow fibre membranes, which allowed automatic cleaning procedures with backwashing and chemical-enhanced cleaning (CEC).

The novel u-t UF membrane was developed using layer-by-layer technology and consisted of hollow fibres with a diameter of 0.8 mm and filtrates from the inside out (Jährgig *et al.* 2018).

The fibre materials were polyvinylpyrrolidone (PVP) and polyethersulphone (PES). A module was 0.2 m in diameter and 1.5 m long, with 12,000 membrane fibres and a membrane area of 40 m² (Figure 2). The technical details of all membrane modules tested are given in Table 1.

2.1.3. Pilot tests

The pilot tests were carried out within two pilot containers (Table 2).

During the pilot tests, the operating parameters such as flux, recovery in relation to the membrane module and crossflow velocity of the pre-treatment membranes were varied, as well as the frequency of backwashing and CECs. For chemical cleaning, caustic soda (NaOH) and sodium hypochlorite (NaOCl) were used first, followed by acid treatment with HCl or citric acid. The chemicals were circulated across the membranes. After soaking, the membranes were flushed. For the cleaning procedure, the ROs were operated with the same settings as during normal operation. Chemical cleaning in place (CIP) was performed manually with NaOH as the first step and HCl as the second step. Table 3 summarises the operational settings during the pilot phase.

2.2. Characterisation of mWWTP effluent

Table 4 shows the quality of the effluent from the secondary clarifier of the mWWTP, which was used as feed water for the pilot plant.

Due to the high fraction of iWW, the electrical conductivity, the concentrations of organic matter (TOC, COD), calcium, hydrogen carbonate and sulphate were two to four times higher than for typical mWW (Levlin 2007; Henze & Comeau 2008; Ho *et al.* 2023). The higher concentrations of organic matter from the pre-treated iWW indicated that this fraction was non-degradable in the mWWTP. The marine eutrophication potential varies widely with ammonium concentrations of 0.01–3.04 mg/L ($n = 32$), nitrate concentrations of 0.4–1.8 mg/L ($n = 27$) and orthophosphate concentrations of 0.1–1.7 mg/L ($n = 10$).

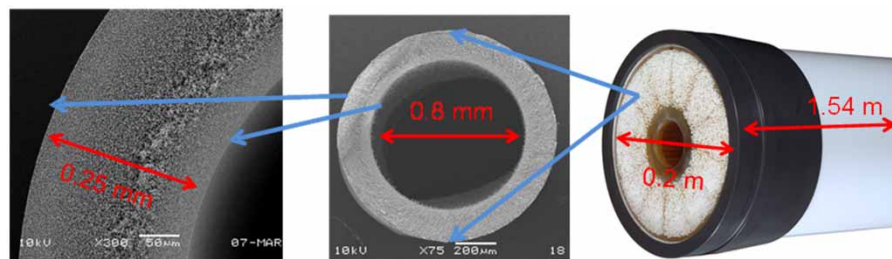


Figure 2 | UF/u-t UF/NF membrane with microscopic details and dimensions of membrane fibres; left: fibre wall, middle: fibre, right: module head.

Table 1 | Technical details of all tested membranes

Parameter	Unit	Conventional UF hollow fibre membrane	Ultra-tight UF hollow fibre membrane	Open NF hollow fibre membrane	RO spiral wound membrane
Name		UFC-LE (Pentair)	Not commercial (Pentair)	HFW1000 (Pentair)	LCHR-4040 (Dupont)
MWCO	kDa	150	4	1	<0.2 ^a
Pore-size	nm	5–20	10 ^a	1–10 ^a	0.1–1 ^a
Material		PVP and PES	Modified PES and PES	Modified PES and PES	
Membrane area	m ²	40	40	40	8.7
Design flux	L/(m ² h)	60–120	20–60	15–30	22.3
Design recovery	%	65–85 (crossflow and dead-end with bleed) (95–99; (crossflow) dead-end)	70–90 (crossflow with bleed)	70–90 (crossflow with bleed)	
Max. system pressure	bar	3	6	6	41
Max. TMP	bar	1	6	6	
Δp	bar		0.15	0.15	1

TMP, transmembrane pressure; PVP, polyvinylpyrrolidone; PES, polyethersulphone.

^aEstimation based on Poirier *et al.* (2023) and Lenntech (2025).

Table 2 | Test set-up for investigations to prevent fouling of the RO membrane

Period	1	2
Pilot A	u-t UF + RO	UF + RO
Pilot B		NF + RO

Table 3 | Tested operational settings of UF, u-t UF, NF and RO

Parameter	Unit	Conventional UF	Ultra-tight UF	Open NF	Parameter	Unit	RO
Flux	L/(m ² h)	50; 60; 65	20; 25; 30; 32.5; 35	20; 22.5; 25; 30; 32.5	Flux	L/(m ² h)	22.3
Water recovery	%	90; 100	75; 80	50; 75; 80	Water recovery	%	40
Crossflow velocity	m/s	0.4	0.3; 0.4; 0.5	0.3; 0.4	Permeate flow	L/h	195
Filtration time	min	20; 25; 30	40; 60	30; 60; 120	Concentrate flow	L/h	300
CEC frequency	1/d	1–7	1–4	0.5–1	Recirculation flow	L/h	1,200
					Antiscalant dosage	mg/L	7.4

2.3. Sampling and laboratory analyses

Samples were taken from the sampling point shown in Figure 1 as grab samples. Samples were collected in a bottle (approximately 2 L) and distributed directly to individual analysis bottles in the pilot plant. They were then picked up by the Eurofins Denmark laboratory. Sampling was carried out regularly, but only when the systems were in stable operation: once per month with a large number of analyses (e.g. aluminium, calcium, carbonate hardness, chloride, copper, conductivity, iron, magnesium, pH, sulphate, TDS, total hardness, TSS, turbidity, *Escherichia coli*, *Legionella*) and weekly sampling with a smaller number of analyses (e.g. COD).

The samples were analysed by Eurofins Denmark using the following methods: aluminium, calcium, copper, iron, magnesium, total hardness: DS 259:2003, DS/EN ISO 17294m:2016 ICP-MS; carbonate hardness was calculated; *E. coli*: DS

Table 4 | Characterisation of mWWTP effluent

Parameter	Unit	Content	n
Electrical conductivity	µS/cm	2,300–6,200	25
Total suspended solids	mg/L	1–17	39
TOC	mg/L	14–50	29
COD	mg/L	40–160	34
Calcium	mg/L	85–240	28
Hydrogen carbonate	mg/L	530–1,300	26
Sulphate	mg/L	280–610	27

TOC, total organic carbon; COD, chemical oxygen demand.

2255:2001; *Legionella*: DS 3029:2001; chloride: DS ISO 15923-1:2013; sulphate (lower concentrations): EN ISO 10304-1 IC-EC; sulphate (higher concentrations): DS ISO 15923-1:2013; TSS DS/EN 872:2005; turbidity: DS/EN ISO 7027-1: 2016; conductivity: DS/EN 27888:2003; pH: DS/EN ISO 10523:2012; COD: ISO 15705; TDS: SS-EN 15216:2021. The operating parameters (flow, pressure, temperature, pH, conductivity, UV254, colour (Pt/Co)) were recorded with the online measurements of the pilot plants.

2.4. Life cycle assessment

2.4.1. Goal and scope

The aim of this LCA was to analyse the potential environmental impacts of the different membrane systems as a tertiary treatment to a mWWTP with the aim of reusing the water for industrial cooling. In detail, the following aspects were analysed:

- The different pre-treatment membranes (UF, u-t UF, NF) before an RO membrane in comparison;
- an assessment of the carbon footprint of different strategies to prevent fouling on the RO, either through biocide dosing or UV treatment of the feed to the RO;
- treatment, concentration and finally evaporation of the RO brine if discharge to the Great Belt is not permitted. This option includes heat recovery from the effluent via a heat exchanger and a subsequent heat pump.

The function of the system under study was ‘to provide wastewater treatment’ including all processes related to this function. The functional unit of this LCA was defined via the annual organic load of the WWTP calculated in population equivalents (pe) of the WWTP ($[(pe\ a)^{-1}]$). To illustrate the product footprint in a perspective of changes to the current WWTP (baseline) in contrast to other water resources, the impact was shown per m³ of product water produced ($[m^{-3}]$).

As this LCA analyses the entire wastewater treatment system, the system boundary includes the entire mWWTP, including its novel tertiary treatment (see Figure 3). Freshwater from Lake Tissø or freshwater from seawater desalination were considered as alternative freshwater resources to water reuse. The operation of the heat exchanger to recover heat from wastewater and reuse it for residual brine treatment was included in one of the scenarios. Finally, the system boundary included background processes for electricity, chemicals, fuels and construction materials for the membrane system. Transport, valorisation of sludge and other solids were excluded and not considered in the LCA.

The specific scenarios in the system perspective are listed below:

- Cooling water from **Lake Tissø**
- Cooling water from **seawater desalination**
- **UF** – coagulation, dual media filtration, UF and RO of secondary effluent including biocide dosing in RO feed, backwash of UF recycled to secondary treatment, RO brine discharged to the Great Belt (south-west Baltic Sea).
- **UF-UV** – coagulation, dual media filtration, UF and RO of secondary effluent including UV disinfection of RO feed, backwash of UF recycled to secondary treatment, RO brine discharged to the Great Belt.
- **u-t UF** – coagulation, dual media filtration, u-t UF and RO of secondary effluent including biocide dosing in RO feed, backwash of UF recycled to secondary treatment, RO brine discharged to the Great Belt.
- **NF** – coagulation, dual media filtration, NF and RO of secondary effluent including biocide dosing in RO feed, backwash of NF recycled to secondary treatment, RO brine discharged to the Great Belt.

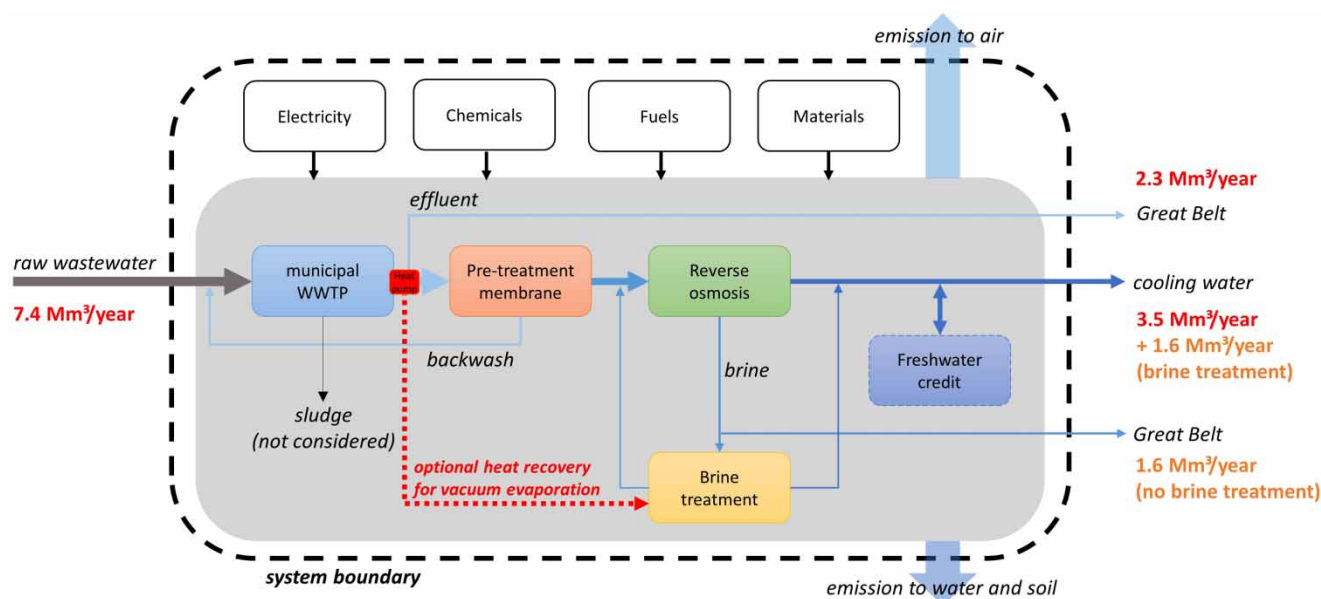


Figure 3 | System boundary and scope of the LCA study Kalundborg WWTP.

- **UF-brine treatment** – coagulation, dual media filtration, UF, RO and brine treatment without heat recovery. RO brine is filtered and then further concentrated via four additional RO stages, the final concentrate is evaporated in a vacuum evaporator.
- **UF-brine treatment – heat recovery** – coagulation, dual media filtration, UF, RO and brine treatment with heat recovery (heat exchanger and heat pump). Similar to the previous scenario, however, it is assumed that the heat required in the vacuum evaporation unit is supplied by a heat exchanger recovering heat from the mWWTP effluent and a heat pump using the recovered heat.

As impact indicators, the global warming potential (GWP) for a time horizon of 100a (IPCC 2023) and a simplified direct water availability footprint (dWAF) using the AWARE scarcity factors (Boulay *et al.* 2018) were considered in this paper.

2.4.2. Input data

For lifting and aeration of Lake Tissø raw water, 0.21 kWh/m³ was estimated (Wendler *et al.* 2022). For seawater desalination, a total water recovery of 45% was assumed (Remy *et al.* 2022). The electricity demand for desalinated seawater was assumed to be between 1.5 and 2 kWh/m³ of freshwater, considering the low salinity of the Baltic Sea. In this LCA an average electricity demand of 1.75 kWh/m³ freshwater was used. Regarding the chemicals for the freshwater supply, the following consumption was assumed for seawater desalination: 10.7 g NaHSO₃ (98%)/(m³ seawater), 41.5 g CO₂ (liquefied 98%)/(m³ seawater), 1.3 g citric acid (100%)/(m³ seawater), 13.8 g NaOH (30%)/(m³ seawater), 35.3 Ca(OH)₂ (92%)/(m³ seawater) and 0.3 g FeCl₃ (40%)/(m³ seawater). Disposal of the resulting iron hydroxide sludge was also considered (Remy *et al.* 2022).

The inventory of electricity and chemicals for the different membrane schemes for water recovery from wastewater is shown in Table 5. The dual media filter had a recovery rate of 96% and a low electricity demand (0.03 kWh/m³). The pre-treatment membranes differed significantly in their recovery rates (71–87%) and electricity consumption (0.08–0.16 kWh/m³). The consumption of chemicals also varied by a factor of two between the different membranes. The low recovery of the u-UF and NF membranes deserves special attention. The high volume of backwash water due to the low recovery rate increases the hydraulic load to the WWTP if this water is not disposed of separately within the Great Belt. The RO membrane had a lower recovery rate (68%) and higher electricity consumption 0.67 kWh/m³. In the scenario ‘UF-UV’, an UV unit (0.04 kWh/m³) was integrated to treat the UF filtrate to avoid biocide dosing in the RO feed. CIPs of the ROs were rather negligible in terms of quantity.

For the brine treatment at first, a filtration step was considered to remove potential precipitates such as calcite, magnesite and silicate from the brine. Thereby it was assumed due to the high carbonate concentrations, that almost all bivalent cations

Table 5 | LCA inventory data of pre-treatment and reverse osmosis

Parameter		Unit	UF-UV	UF	u-tUF	NF
Sand filter	Recovery	%		96		
	Electricity	kWh/m³ feed		0.03		
	FeCl₃ (40%)	g/m³ feed		5		
Pre-treatment membrane	Recovery	%	87	87	73	71
	Electricity membrane	kWh/m³ feed	0.08	0.08	0.09	0.16
	NaOH (28%)	g/m³ permeate	4.5	4.5	6.2	2
	NaOCl (12%)	g/m³ permeate	1.4	1.4	2.2	1.7
	CitricA (40%)	g/m³ permeate	9.5	9.5	14.8	10.6
	Electricity UV	kWh/m³ feed	0.04			
RO	Recovery	%		68		
	Electricity	kWh/m³ feed		0.67		
	Biocide (20%)	g/m³ feed	-		4	
	NaOH (35%)	L/CIP		83 (5.5 CIP/a)		
	HCl (25%)	L/CIP		144 (5.5 CIP/a)		

causing scaling were removed with carbonate and mono-valent high soluble cations remaining in solution. The residual brine had then to be further concentrated by at least a factor 10 using RO membranes. The specific chemical consumption for the CIPs was assumed to be similar to that for the pilot ROs (Table 5). Recovery and electrical consumption were calculated using WAVE software (DuPont 2020). Through several concentration steps with RO membranes an overall recovery rate of 89% and a specific electricity consumption of 1.65 kWh/m³ were obtained with respect to the initial brine. The final brine was assumed to be fed into a vacuum evaporation unit. This evaporates water under low-pressure conditions below 100 kPa and condensates water under normal pressure conditions. This design allows water to be evaporated at 35–40 °C and recovered from the condensate. The heat was assumed to be reused internally, reducing the overall energy demand of the system. The selected system (Veolia 2024) consumes 20 kWh_{el}/m³ of electricity and 300 kWh_{th}/m³ of external heat, recycling approximately 50% of the energy required for water evaporation internally. To reduce the heat demand, an existing heat exchanger in the WWTP could be recommissioned together with an existing heat pump. This might provide sufficient heat as required; but the heat pump consumes additional electricity. A coefficient of performance of 4 was assumed (Qian 2010), e.g. a consumption of 75 kWh_{el}/m³ distillate or 3.4 kWh_{el}/m³ product water was used in the calculation.

3. RESULTS AND DISCUSSION

3.1. Performance of UF, u-t UF and NF: removal of selected parameters

The removal efficiencies of the tested UF, u-t UF and NF membranes are shown in Figure 4 for selected parameters such as turbidity, chemical oxygen demand (COD), total organic carbon (TOC), sulphate, total hardness and electrical conductivity.

Turbidity retention was between 90 and 95% for all three membranes. The COD, TOC and sulphate retentions were 73, 50, and 25%, respectively, and higher for the NF membrane compared to the UF and u-t UF membranes with approximately 25, 15 and 5% and below. The removal capacities for total hardness and electrical conductivity were only slightly different with 15 and 5% for the NF and 5 and 3% for the UF and u-t UF, respectively.

Turbidity removal was high for all membranes, as expected. The NF retained the organic compounds better than the UF and the u-t UF, which was also expected because the lower MWCO of the NF (1 kDa) compared to the u-t UF (4 kDa) and the UF (150 kDa) leads to better retention. Surprisingly, although the MWCO of the u-t UF was smaller than that of the UF, the retention of TOC was in a similar range for both. One reason may be the very small size of the organic compounds, which allows them to pass through pore sizes with an MWCO of 4 kDa. Ezugbe & Rathilal (2020) and Poirier *et al.* (2023) show a higher retention of COD, but due to the iWW fraction coming from the biotech and pharmaceutical industry, it is likely that very small organic compounds were present in the wastewater mix. This was also observed by Alturki *et al.* (2012). Therefore, their low molecular weight allowed them to pass through the u-t UF membrane. In conclusion, the NF performed much better than the UF and u-t UF. However, the advantage of the u-t UF with a lower MWCO compared to the UF was very small and showed only negligible or slightly better removal rates.

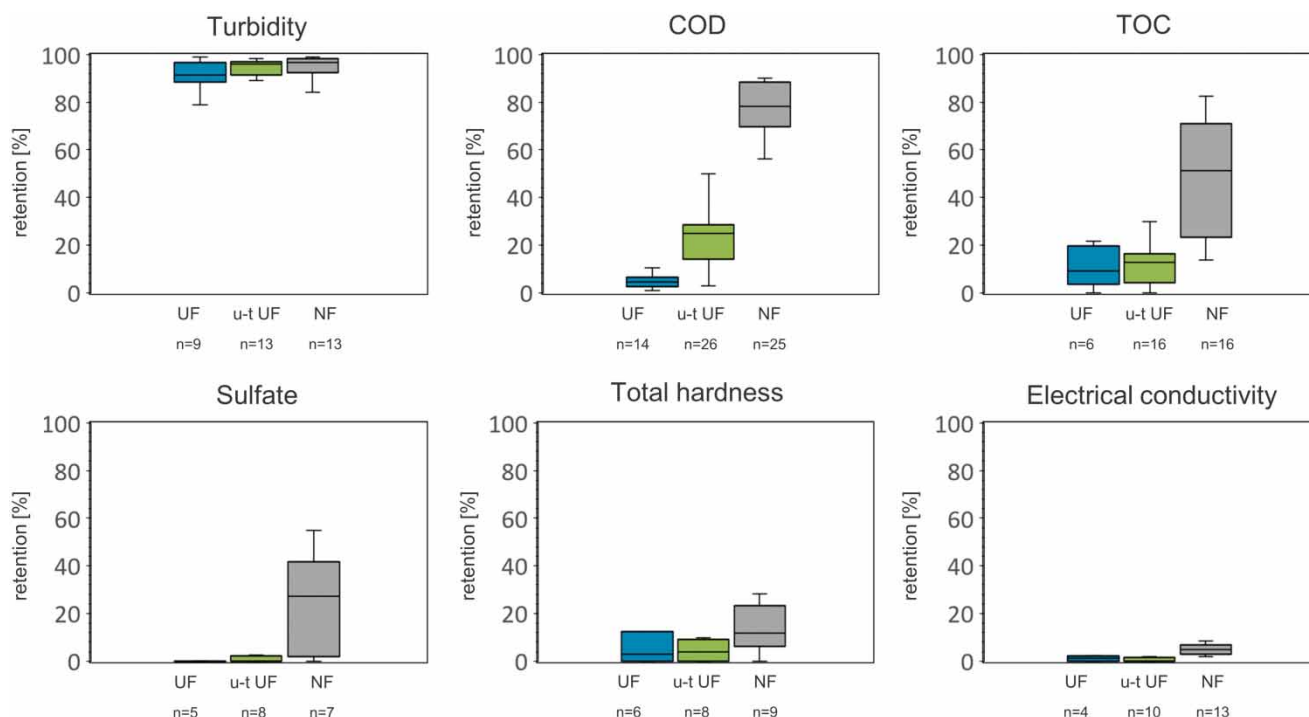


Figure 4 | Performance of UF, u-t UF and NF: removal of selected parameters with number of samples; all analysis from 2 years of operation, independent from operational settings, dosing and pre-treatment.

3.2. Permeability of RO after UF, u-t UF and NF: Protection in terms of fouling

The high concentrations of organic matter (TOC, COD), calcium, hydrogen carbonate and sulphate in the feed water (see Figure 1) are likely to lead to organic fouling and scaling on the RO membranes, thereby increasing the operating transmembrane pressure (TMP). In addition, the electrical conductivity also indicated a high salinity and, together with the high total suspended solids (TSS) content, an increased RO operating pressure was expected. The increased pressure was expected to lead to a higher energy consumption of the ROs and to require more frequent cleaning procedures, thus increasing operating costs.

In Table 6, the feed water qualities for both ROs are shown for periods 1 and 2.

Figure 5 shows RO permeability as an indicator of RO performance. In the first period, the addition of biocide was avoided to test whether a denser pre-treatment membrane could replace the biocide treatment normally required (Figure 5, left graph). A significant decrease in permeability was observed for both membrane combinations, u-t UF with RO (20% decrease during 11 days of operation) and NF with RO (20% decrease during 18 days of operation). CIPs were used to increase the permeability. However, after each CIP only a small recovery in permeability was observed. In the second period (Figure 5, right graph), biocide was dosed regularly (once a week) into the feed tank of the RO. This kept the RO permeability at a stable level for both pre-treatment options (UF/NF).

Table 6 | Feed water quality of ROs during periods 1 and 2, selected parameter

	COD		Conductivity		Hardness, total		COD		Conductivity		Hardness, total	
	mg/L	n	mS/m	n	°dH	n	mg/L	n	mS/m	n	°dH	n
Period 1	Ultra-tight UF						Open NF					
	51 ± 6	21	525 ± 73	4	29 ± 7	3	15 ± 17	18	503 ± 81	4	26 ± 10	3
Period 2	Conventional UF						Open NF					
	50 ± 6	14	468 ± 33	4	26 ± 8	3	15 ± 6	17	443 ± 38	4	21 ± 4	3

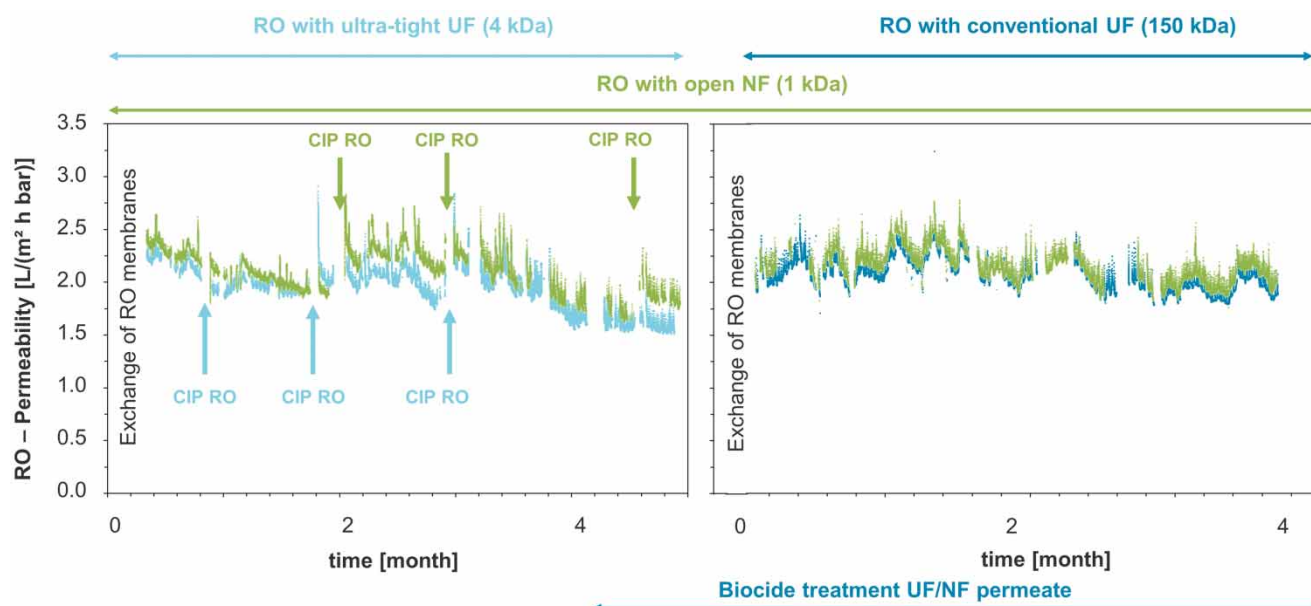


Figure 5 | Permeability of RO with different pre-treatment membranes: (left): u-t UF and NF with CIPs, (right): UF and NF without CIPs, but a weekly biocide treatment of the RO feed.

The decreases in permeability in the first period suggest that fouling processes occurred on the RO membranes of both combinations. The feed water quality of both ROs differed only slightly: the organic content (COD) was on a similar level, the conductivity and the hardness were about 10% higher in the first period compared to the second period. As the CIPs only removed a part of the impurities, irreversible fouling occurred. However, in the second period, no permeability decrease was observed and therefore no fouling occurred as a very likely result of the biocide dosing. Thus, the fouling in the first period was mainly due to microbial activity. As the wastewater was quite warm (approximately 22 °C), even more biofouling was expected. Biocide dosing is the most commonly used technique to prevent biofouling on and in the RO, but it is also very dangerous (Da-Silva-Correa *et al.* 2022). Denser membranes with an MWCO of 4 and 1 kDa as pre-treatment for the RO feed alone could not prevent biofouling under the conditions in this study. However, the biocide had a significant positive effect on the performance of the ROs, while the different pre-treatment membranes showed no clear difference. In addition, biocide dosing eliminated the need for CIPs for four months. This means that as long as the biocide is dosed, the conventional UF membrane is sufficient to maintain stable operation of a RO membrane.

3.3. Comparison of operational parameters: Conventional UF, ultra-tight UF and open NF

Table 7 shows the operating parameters for all membranes tested. The conventional UF could be successfully operated with a flux of 60 L/(m² h) and a recovery of 90–99%, the net recovery including all waste streams was 87%. The u-t UF and the open NF achieved max. 37 L/(m² h) and max. 32.5 L/(m² h) as flux and a recovery of 80% respectively. Their net recoveries of 73 and 71% were similar. The measured TMP ranged between 0.04–0.9, 0.15–1.0 and 1.0–2.7 bar for UF, u-t UF and NF, respectively. The TMP depended on the feed water quality and the operating time after chemical cleaning (CEC) and correlated with the energy consumption of 0.08, 0.09 and 0.16 kWh/(m³ feed) for the UF, u-t UF and NF respectively. The CIP frequencies of the ROs were calculated using the permeability of the ROs. Only the biocide treatment period was used for the calculation. Using the UF as pre-treatment, the RO would need to be cleaned 3–5 times per year and with NF 0–4 times per year.

The highest flux and recovery efficiencies were achieved with conventional UF. For the design of a full-scale system, the high fluxes and recovery rates allow for smaller membrane areas and therefore a lower number of modules to achieve the same amount of reclaimed water. It should be noted that UF produces less wastewater (extremely low concentrate flow, only waste streams from backwash and chemical cleaning). For the conventional UF, the TMP and the corresponding energy consumption were lower than for the other membranes, as expected due to the higher MWCO. This is a major

Table 7 | Comparison of operational parameters

Parameter	Unit	Conventional UF	Ultra-tight UF	Open NF
MWOC	kDa	150	4	1
Flux	L/(m ² h)	50; 60 ; 65	15; 37	15; 32.5
Water recovery ¹⁾	%	90 ; 99	75; 80	50; 75 ; 80
Net recovery ²⁾	%	87	73	71
TMP ³⁾	bar	0.04 – 0.9	0.15 – 1.0	1.0 – 2.7
Energy consumption	kWh/(m ³ feed)	0.08	0.09	0.16
Frequency of CIP RO (with biocide)	1/a	3 – 5	---	0 – 4

Conventional UF (in dead-end mode), u-t UF (in crossflow mode) and NF (in crossflow mode). Bolded values indicate preferred settings; green shade indicates good results, yellow shade indicates medium results, red shade indicates poor results. The superscript number 1 indicates set point recovery for filtration. The superscript number 2 indicates recovery including waste streams. The superscript number 3 indicates for preferred settings; depending on feed water quality and time after CEC.

advantage. The calculated frequency of CIPs was only slightly higher for the RO with UF as pre-treatment, which is not a major disadvantage.

All membranes tested were suitable for RO pre-treatment. However, the most suitable pre-treatment for a full-scale plant seems to be the conventional UF due to its lower energy demand and higher flux and recovery efficiency. The expected beneficial effects of using more dense membranes such as the u-t UF or NF to reduce RO membrane fouling could not be demonstrated during pilot operation.

3.4. Cooling water quality

The comparison of the water quality of the RO permeate with the required water quality for cooling purposes and the raw lake water, which after aeration is currently used as cooling water, is shown in Table 8. The results refer to the combined permeate from both ROs, regardless of their pre-treatment method. The water quality requirements were defined using technical guidelines (VDI 2047 Bl. 2 2015; Niewersch *et al.* 2016; VDI 3803 Blatt 1 2019) and the Spanish Regulation for Water Reuse (Royal Decree 1620/2007 2007). For Denmark, neither a regulation nor guidelines, dealing with water reuse for cooling purposes were available.

For the concentrations of aluminium, the carbonate hardness, chloride, copper, conductivity, iron, magnesium, sulphate, total dissolved solids (TDS), total hardness, TSS and the abundance of *E. coli* and *Legionella*, the RO permeate met the required water quality. However, for calcium and pH, the permeate water quality was actually below the required thresholds. The water quality parameters of the RO permeate were lower than the raw lake water for all parameters.

Higher levels of calcium and pH are required. This can easily be achieved with a common post-treatment process such as a chemical stabilisation. Full compliance with all requirements of the technical guidelines and the Spanish regulation shows that all treatment trains were suitable to produce the required water quality. Niewersch *et al.* (2016), Van Houtte & Verbaudhede (2012, 2013) and Nahrstedt *et al.* (2020) made similar observations, and also used a UF and RO as well as an NF and RO to successfully produce fit-for-purpose water for industrial use (e.g. cooling). The requirements for the abundance of *E. coli* and *Legionella* were already achieved in the permeate of the UF and NF (<1 FNU/ < 10 CFU/L), which is also typical for both membrane types and is frequently observed in practice (Bodzek *et al.* 2019). Furthermore, all treatment trains produced water of even better quality than that of the currently used water from Lake Tissø. Therefore, the produced fit-for-purpose water is very suitable for reuse as cooling water in the industry.

3.5. Life cycle assessment

3.5.1. Global warming potential

All scenarios were calculated with the electricity mix from Ecoinvent 3.9 for Denmark with a GWP of 0.211 kg CO₂-Eq/kWh_{el}. In terms of the WWTP operation (secondary treatment) only changes to the current baseline were considered (e.g. the impact via backwash from the membranes). Freshwater from Lake Tissø had the lowest GWP (0.04 kg CO₂-Eq/m³) due to the very simple treatment with lifting and aeration (see Figure 6). Desalinated seawater had the highest impact

Table 8 | Quality of RO permeate and comparison with cooling water quality and currently used lake water quality

Parameter	Unit	Limit for cooling water ¹⁾	RO ²⁾	n	Raw lake water	n
Aluminium	µg/L	< 500	< 44 ³⁾	38	n.a.	
Calcium	mg/L	> 20 / < 500	< 0.5	23	93	2
Carb. hardness	°dH	< 4 / < 20	< 0.1	16	n.a.	
Chloride	mg/L	< 50 / < 250	13.4	25	39	3
COD	mg/L	< 5	< 5	30	30.3	3
Copper	µg/L	< 500	< 0.5	25	3.8	3
Conductivity	µS/cm	50 – < 3000	5.8	29	550	3
Iron	mg/L	< 0.1 / < 0.5	< 0.05	19	0.1	3
Magnesium	mg/L	< 100	0.1	24	8.3	2
pH		7 – 9	6.4	28	8.4	3
Sulfate	mg/L	< 50 / < 600	1.0	24	67	3
TDS	g/L	< 1.8	0.06	21	n.a.	
Total hardness	°dH	0.1 – < 8	< 0.1	23	14.7	2
TSS	mg/L	< 5	0.6	18	6	3
Turbidity	FNU	< 1	< 1 ³⁾	48	1.8	3
<i>Escherichia coli</i>	MPN/100 mL	Absence	< 1	19	2.8	3
<i>Legionella</i>	CFU/L	< 100	< 10	11	n.a.	

Green shade indicates cooling water quality reached/lake water quality exceeded, yellow shade indicates cooling water quality can be reached with common post-treatment. The superscript number 1 indicates Royal Decree 1620/2007 2007; VDI kBl. 2 2015; VDI 3803 Blatt 1 2019; Niewersch *et al.* 2016. The superscript number 2 indicates independent from pre-treatment, results of both ROs together. The superscript number 3 indicates already reached after UF/NF.

with 0.57 kg CO₂-Eq/m³, excluding the brine treatment scenarios. The main contributor for reuse was the electricity demand, especially for the RO membrane. The water reuse scenarios were in between with 0.33–0.40 kg CO₂-Eq/m³, with both UF scenarios being the most favourable in terms of GWP. The brine treatment significantly increased the GWP. The water evaporation of the concentrated brine was the main energy driver, either due to the heat demand if no heat recovery was installed, or due to the electricity demand for the heat pump.

In terms of lake water treatment, Wendler *et al.* (2022) calculated 0.13 kg CO₂-Eq/m³ for a similar treatment, however, the distinction can be ascribed to the different electricity mix under study. Regarding seawater desalination, it should be underlined that the Great Belt has hereby a quite low salinity compared to the Mediterranean Sea, the North Sea or the Atlantic Ocean. Consequently, also the electricity demand (1.5–2 kWh/m³ produced water) impacting the GWP was severely low for a seawater desalination plant. In previous studies, an electricity demand of 4 kWh/(m³ produced water) (Lattemann 2010) was assumed to lead to 2.56 kg CO₂-Eq or 1.90 kg CO₂-Eq depending on the electricity mix (Kraus *et al.* 2016). Remy *et al.* 2022 assumed an electricity demand of 2.65 kWh/(m³ produced water) leading to a footprint of 1.33 kg CO₂-Eq/m³. The different results regarding the inventory can be ascribed to developments, different salinities and different electricity mixes per country and year. Nonetheless, the GWP is still higher compared to reused wastewater, although this wastewater was highly influenced by iWW and had therefore a quite high salinity for wastewater.

For the membrane treatment of WWTP effluent in this study we assumed 0.08 kWh/(m³ feed) for the UF with 87% recovery, 0.09 kWh/m³ feed for the u-t UF with 73% recovery, 0.16 kWh/m³ for the NF with 71% recovery. The recovery depended on the DOC and Fe-dosage. the higher the Fe-dosing, the higher the recovery, as shown by Mieke *et al.* (2014). The RO consumed 0.67 kWh/(m³ feed) at 68% recovery. A full-scale RO with 77% recovery and 0.75 kWh/(m³ feed) and a large pilot plant with 85% recovery and 0.71 kWh/(m³ feed) were operated in Torreele (Belgium) and in Shafdan (Israel), respectively (Kraus *et al.* 2016). Based on these values, the reported GWP for the Torreele system (but with submerged UF membrane) was 0.37 kg CO₂-Eq/(m³ produced water) and very similar to the footprint of this study with UF membranes of 0.32 kg CO₂-Eq/(m³ produced water), while for the Shafdan system the GWP was significantly higher at 1.30 kg CO₂-Eq/m³. The similarity with Torreele and the

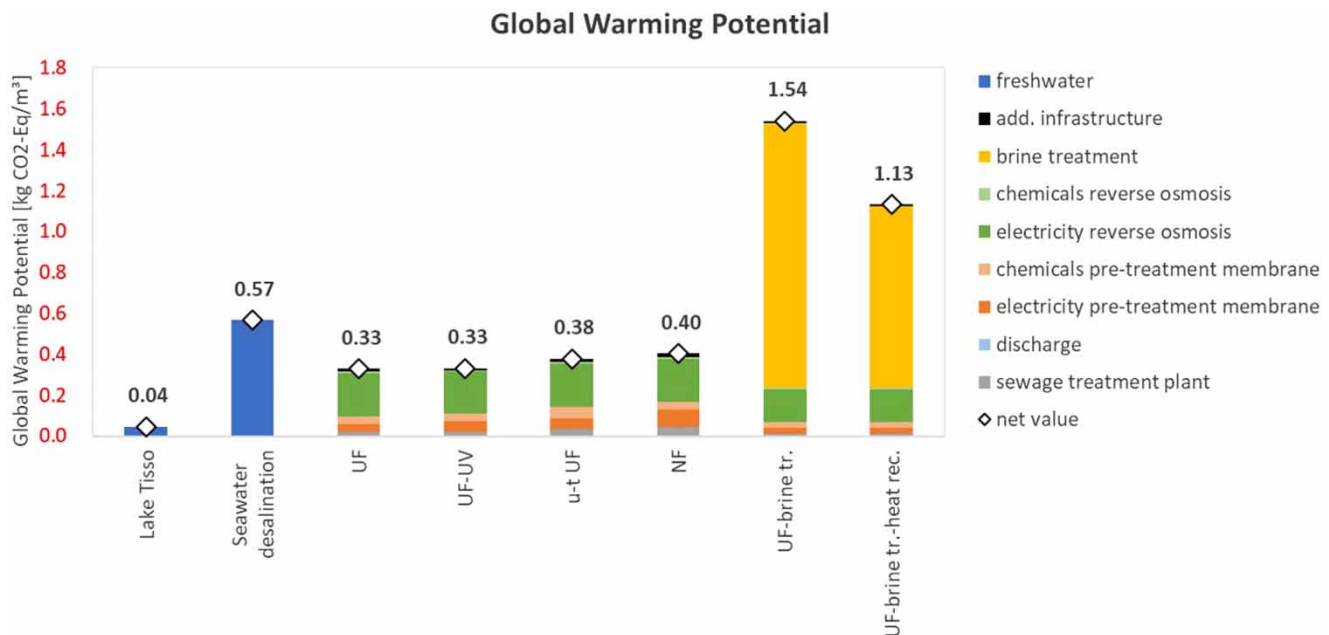


Figure 6 | Global warming potential (product perspective) of different freshwaters and reused waters for industrial cooling purposes.

difference with Shafdan can be explained by the different electricity mix per country, with Denmark and Belgium having quite similar values for CO₂-Eq/kWh compared to Israel.

3.5.2. Marine eutrophication potential

In terms of marine eutrophication potential, the benefits of the water reuse system were evident, with less nitrogen being discharged to the Great Belt (see Figure 7). In the scenarios without brine treatment, the backwash from the dual media filter and the pre-treatment membrane was returned to secondary treatment, with partly nitrification and denitrification of the

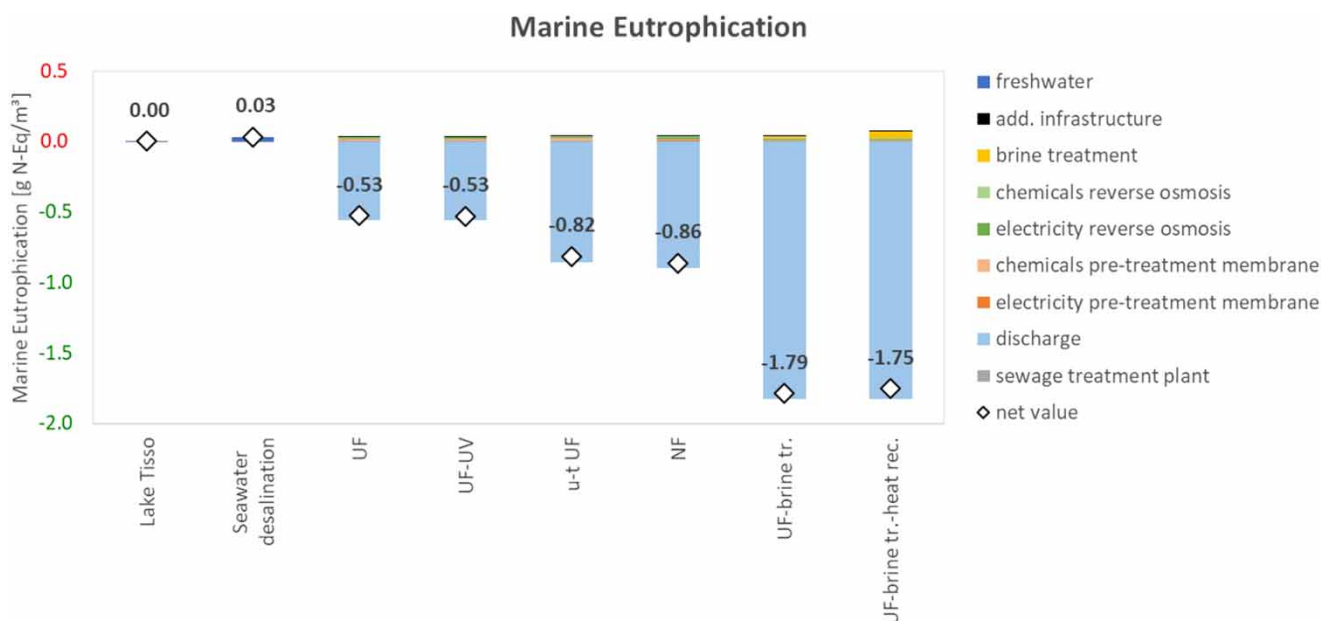


Figure 7 | Marine eutrophication potential (product perspective) of different freshwaters and reused waters for industrial cooling purposes.

nitrogen in this stream. With brine treatment, even less nitrogen was discharged to the Great Belt, resulting in higher absolute and relative benefits in terms of marine eutrophication potential.

The overall impact on marine eutrophication was highly dependent on the nitrogen load of the specific WWTP. However, Kraus *et al.* 2016 showed benefits within this impact category in combination with membrane systems, i.e. such systems remove nitrogen from the effluent and, depending on backwashing and brine management, this removed nitrogen is (partially) eliminated.

3.5.3. Direct water availability footprint

The freshwater abstraction from Lake Tissø had a dWAF of 2.03 m³-Eq/m³ due to the water scarcity factor of 2.03 from the AWARE model for Denmark. For all other scenarios, the dWAF was zero, as treated wastewater was either discharged into the Great Belt or cooling water was obtained from wastewater or seawater, which does not consider any water withdrawal or release from the freshwater environment. Therefore, our preferred configuration of a dual media filter combined with UF and RO is also very suitable from a dWAF perspective of zero.

4. CONCLUSIONS

In order to identify the best configuration for a potential water reclamation plant, pilot studies and an LCA based on the up-scaled pilot results were carried out. The water reclamation pilot plant was applied to the effluent of a WWTP that also treats pre-treated iWW from a biotech industry.

Different pre-treatment membranes such as a UF, u-t UF and NF were tested to determine the best option to prevent organic fouling and scaling in a subsequent RO module:

- Only minor differences between the RO protection efficiencies of the tested pre-treatment membranes were observed suggesting that RO can be operated with all pre-treatment membranes.
- No clear advantages of the newly developed u-t UF were found. Flux and recovery were similar to open NF but COD and TOC retention was not as high as expected.
- Biocide dosage, together with CIP frequency, had the greatest effect on RO performance.
- All membranes were able to handle variable feed quality.
- The settings tested showed the highest recovery rate for the conventional UF. This means that fewer modules are required for a full-scale plant. When operating in dead-end mode, the UF will produce less wastewater. These are clear advantages of the conventional UF.
- The advantages of a denser membrane (u-t UF, NF) are that the permeate can be used as a low-grade process water with better water quality in terms of turbidity, organic matter content and colour compared to conventional UF.

A life cycle assessment of the up-scaled pilot system was carried out and the following results were obtained:

- Water reclamation, as well as freshwater supply from seawater desalination, can reduce freshwater withdrawal from freshwater environments.
- Only if the use of freshwater from Lake Tissø is no longer possible, there will be benefits from using reclaimed water instead, as shown in the direct water availability footprint.
- Water recovery is associated with a higher energy demand and carbon footprint if the recovered water replaces freshwater from Lake Tissø, while it is associated with a lower energy demand and carbon footprint if the recovered water replaces water from seawater desalination.
- Of all the pre-treatment membranes, conventional UF had the lowest footprint and the highest recovery rate and was therefore the preferred option.
- In a full-scale system, the recovery rate and therefore the backwash volume is of high importance due to the hydraulic limitation of the secondary treatment.
- A combination of conventional UF and UV disinfection showed a similar overall footprint to the use of biocide in the RO feed and could be advantageous as no residual biocide is discharged with the RO brine to the Great Belt.
- The brine treatment to zero liquid discharge was very energy intensive, in particular the water evaporation of the final brine after several mechanical concentration steps significantly increased the energy demand and the carbon footprint of the entire reuse system. Although a heat recovery system with a heat pump was able to reduce the energy demand and carbon footprint, the brine treatment to zero liquid discharge remained energy intensive.

- Finally, the water reclamation scheme with pre-treatment membrane and RO reduced the total phosphorus and nitrogen loads to the Great Belt, although the nitrogen concentrations in the RO brine discharged to the Great Belt increased compared to the secondary effluent.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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