ULTIMATE

WATER SMART INDUSTRIAL SYMBIOSIS

Deliverable D1.3

New approaches and best practices for water recycling in symbiosis cluster

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Technical References

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[1] PU = Public

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Executive Summary

Summary of Deliverable

The European Horizon 2020 ULTIMATE promoted circular economy concepts within the framework of water smart industrial symbioses. It focused on industrial wastewater streams as resource for water, energy and material recovery. ULTIMATE, in nine case studies, developed and demonstrated 24 circular economy related technologies. In seven out of the nine case studies, a total of eleven water-related technologies were investigated.

D1.3 focuses on wastewater treatment and reuse technologies in various European regions and contributes to building a circular water economy. By promoting efficient wastewater management and reducing reliance on freshwater resources, it supports industries, and agriculture in some cases, while preserving natural ecosystems.

Most of the lessons learned outlined in this document focus on the more technical and scientific aspects of the proposed technologies in the various case studies, although economic and regulatory aspects are also discussed. This is why the findings presented in this report also aim to contribute to shape policies, drive innovation, and foster cooperation, ultimately promoting sustainable practices that benefit both the environment and the economy across Europe.

Water recovery and reuse

The **case study in Tarragona (CS1)** aims to improve water availability in a petrochemical complex by over 20% through enhanced water recycling. This involves treating effluent from an industrial wastewater treatment plant (iWWTP) using a near-zero liquid discharge (nZLD) process based on membranes. The process includes ultrafiltration (UF), reverse osmosis (RO), and membrane distillation (MD). UF removes turbidity (86%) and reduces total suspended solids (TSS) and chemical oxygen demand (COD) by 51% and 18%, respectively, with an average energy consumption of 0.7 kWh/m³. RO is used in two passes to meet quality requirements, reducing conductivity and ammonium, achieving a global recovery rate of 20-30%. The treatment of the RO concentrate by MD has not yielded good results regarding the quality of the permeate obtained, which does not meet the required limits for reclaimed water in terms of conductivity and ammonium concentration. Further testing is needed to optimize the operation (feed pH adjustment) and verify that a higher quality permeate can be achieved to be reused.

An alternative approach, using zeolite adsorption to replace the second RO step of the water reclamation plant (WRP), was analysed, which could lower energy consumption by 6% and improve water recovery. Currently, it could not be considered a viable alternative since, although it has proven capable of adsorbing ammonium, it does not

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reduce conductivity; therefore, the output stream would not meet the required quality standards.

In **the Dutch case study (CS2)**, wastewater from greenhouses is treated with electrodialysis (ED) and capacitive electrodialysis (CED) to reduce salinity and produce irrigation water with low conductivity (<0.2-1 mS/cm) and low sodium and chloride levels (<0.1 mmol/L). ED effectively removes monovalent ions like chloride and nitrate, achieving approximately 90% reduction in electrical conductivity, although divalent and trivalent ions (e.g., sulfate, calcium, magnesium, phosphate) are removed more slowly. The system shows strong performance for monovalent ion removal, with specific energy consumption (SEC) of 0.12-0.14 kWh/m³.

(C)ED demonstrated high-quality irrigation water across different feed types, with variable ion removal efficiencies. Sodium removal was lower than that of calcium and magnesium (86%, 97%, and 98%, respectively, for target conductivity <0.2 mS/cm), influenced by voltage and water recovery. (C)ED's energy efficiency and customizable ion removal make it a promising sustainable alternative to traditional desalination, especially for irrigation water and nutrient recovery in greenhouse horticulture.

Overall, (C)ED shows potential as a sustainable solution for water reclamation from greenhouse wastewater, achieving 60-90% water recovery and supplying reclaimed water equal to 16% of the consortium's total freshwater use.

In the **Italian case study (CS3)**, an industrial symbiosis was established between an industrial company and a water utility to reuse urban wastewater for industrial purposes, which requires low-salinity water. However, the area faces saline intrusion issues, creating water conflicts between various economic activities. To mitigate this, a digital solution was proposed, using a dynamic simulation model to predict seawater infiltration and incorporating digital tools like a smart equalization system and a decision-support matchmaking platform. This system forecasts salinity peaks in the sewage networks of the municipalities of Cecina and Rosignano and allocates wastewater and reclaimed water for industrial, agricultural, or specific treatments (e.g., reverse osmosis) to optimize reuse.

The storm water management model (SWMM) models effectively predicted the quantity and quality of seawater infiltrating the sewage networks, showing that salinity in wastewater exceeded industrial reuse limits. The smart equalization system enabled a 19% discharge to reduce salinity by 23%, with 87% of reclaimed water directed to industry and 13% to agriculture. For agricultural reuse, crop tolerance to water conductivity was considered to determine suitable reuse levels.

As the equalization system alone couldn't meet the industrial reuse quality standards, a reverse osmosis (RO) unit was piloted to improve water quality. This pilot, fed by the pre-UV effluent from ARETUSA, produced high-quality permeate with very low conductivity, allowing for dilution of high salinity reclaimed water to meet industrial

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requirements. However, the treatment cost of 0.33 €/m³ was high, suggesting limited use for specific needs.

In the **Greek case study (CS4)**, wastewater from an orange juice processing plant is treated to recycle water for irrigation and recover polyphenols as valuable by-products. The wastewater is processed with VesperX, a system that combines physicochemical and biological methods to isolate valuable compounds and render the water suitable for irrigation or industrial reuse.

Various technologies were tested. In the VAC adsorption/extraction module, 80% of phenolic compounds were selectively adsorbed onto resin, removing around 20-25% of TOC. Following polyphenol removal, coagulation was used to eliminate suspended solids and 50% of TOC. The water, pH-adjusted, was then treated with Advanced Oxidation Processes (AOP) using CPC and annular photocatalytic reactors. A small bioreactor platform (SBP) was also employed when needed. AOP and SBP together improved efficiency, reducing time and cost.

The solar AOP reactor did not reduce TOC as expected, likely due to organic molecules fragmenting without full mineralization to CO_2. However, in the annular AOP reactor, H_2O_2 was used to avoid complex catalyst recovery, resulting in complete degradation of a model compound and 50% mineralization within 30 minutes.

In the **second Spanish case study in Lleida (CS5)**, the effluent of the brewery wastewater treatment plant is proposed to be further treated by nanofiltration in combination with reverse osmosis in order to produce fit-for purpose water to be reused in cooling towers. The quality to be achieved is under the framework of an existing national regulation Royal Decree 1620/2007. Additionally, salinity has to be removed from the wastewater for preventing scaling in cooling towers. The combination of a nanofiltration (NF) with a molecular weight cutoff of 1kDa is used as pre-treatment for the reverse osmosis (RO). The KPI reduction of fresh water through re-use of treated wastewater >10% has been reached. Water consumption in brewery is ca. 1400 m^3/d, and water reuse potential (only for the studied cooling towers use) can be as high as >15% of daily needs, which could potentially be provided by the proposed solution.

In the **Tain case study in Scotland (CS7)**, the current effluent treatment plant (with screens followed by an anaerobic membrane bioreactor, AnMBR) enables the production of high-quality water that can be reused in the distillery for cleaning or cooling. However, the AnMBR effluent still contains high levels of nitrogen, phosphorus, some organic compounds, and salinity. A treatment sequence to remove and recover nutrients was studied, involving struvite precipitation, ammonia stripping, and reverse osmosis (RO) membranes to remove other contaminants and salinity.

Three sequences were tested: in the first, the RO membranes were placed directly after the AnMBR; in the second, struvite precipitation was applied first to reduce fouling on the membranes; and in the third, both nutrient recovery technologies were used

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before the RO membranes. The results showed that RO membranes effectively removed contaminants in all sequences: conductivity was reduced by more than 95%, COD by 99%, phosphorus by 99%, and other ions by at least 96%. However, only ammonia exceeded the quality criterion for cooling water (1 mgN/L).

The salinity and contaminants in the effluent affect the operating conditions of the RO system due to osmotic pressure and fouling propensity. Implementing nutrient recovery systems before the RO increased salinity and, consequently, fouling, due to the high pH required for the reactions. The results suggest that applying RO directly after the AnMBR and then the recovery systems in the concentrate stream could be more cost-effective (sequence 1).

Finally, the key performance indicator was the reduction of freshwater consumption by reusing 40% of the treated effluent. At full scale, with a 50% recovery from 322 m^3/day of wastewater, up to 66% of tap water consumption could be covered, representing a significant reduction.

Finally, the **Danish case study in Kalundborg (CS9)** focused on treating effluent from a municipal wastewater treatment plant (mWWTP) to produce water suitable for industrial reuse, like cooling or steam production. The treatment process tested combined ultrafiltration (UF) or nanofiltration (NF) with reverse osmosis (RO). In two pilot plants, three types of membranes were tested: a standard UF membrane, a novel ultra-tight UF membrane, and a conventional open NF membrane, all followed by RO. Additional pre-treatment included a Dynasand and dual media filter.

Results showed that all membranes achieved high turbidity retention (90-95%), but the NF membrane had the best retention rates for COD, TOC, and sulfates. However, the conventional UF membrane offered the highest flux and recovery rates, making it the most energy-efficient option for upscaling. The UF-RO combination was found to be the best configuration due to its robust handling and lower membrane requirements, producing reclaimed water quality even better than raw lake water. A full-scale plant is planned to replace lake water for cooling, with an expected production of 3.5 million m^3/year.

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CS	Technology	TRL	Water type produced or saved	Inflow flowrate/ capacity	KPI
1	UF+RO+MD	5→7	Cooling water	0.5-1 m^3/h (UF) 22-49 m^3/h (RO) 375-545 L/h (MD)	✓ Reduction of fresh water through re-use of treated wastewater >20%
1	Ammonium adsorption on zeolite	5→6	Cooling water	150-250 L/h	✓ 10% energy consumption savings
2	ED/(C)ED	6→7	Irrigation water	1-4 m^3/day	✗ Reduction of fresh water through re-use of treated wastewater >20%
3	SWMM+RO	5→8	Irrigation water or industrial water, depending on the quality	450-800 L/h	✓ Reduction of fresh water through re-use of treated wastewater >35%
4	VAC adsorption/extraction +coagulation+AOP reactors+Small bioreactor platform	6→7	Irrigation water	10 m^3/day	✓ Reduction of fresh water through re-use of treated wastewater >90%
5	NF+RO	7→9	Cooling water	0.8-1.6 m^3/h	✓ Reduction of fresh water through re-use of treated wastewater >10%
7	RO	5→7	Cleaning water and cooling water	0.1 L/s	✓ Reduction of fresh water through re-use of treated wastewater >40%
9	Dynasand/media filter + UF/u-t UF/NF+RO	5→7	Industrial water	0.6-4.8 m^3/h (UF/NF) 0.2 m^3/h (RO)	✓ Reduction of freshwater consumption through reuse of the treated wastewater > 40%

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EU-added value of the deliverable

This report presents an overview of different wastewater treatment technologies designed to enhance water reuse, mainly in the industrial sector, providing valuable data and recommendations for the EU to use in developing or revising environmental policies and regulations. The evidence-based insights offered can help shape new frameworks for water reuse and wastewater management, ensuring alignment with environmental sustainability goals.

The report also provides a solid analytical foundation for the creation of new regulatory standards, such as stricter wastewater quality requirements and more efficient licensing procedures. These insights can help harmonize practices across member states, ensuring consistent high standards for water quality and reuse. It can also aid in aligning national policies with EU-wide objectives, promoting cooperation on cross-border water issues and encouraging a unified approach to water reuse and resource management.

Moreover, the report underscores the need for innovation in wastewater treatment technologies, potentially driving research investments in areas such as membrane filtration, energy-efficient treatments, and decentralized wastewater systems. This could stimulate technological advancements and improve the efficiency of water reuse processes. EU institutions can leverage the findings to inform decisions on funding, research, and infrastructure development for wastewater treatment projects. For instance, the report highlights the benefits of advanced water treatment technologies, which could influence decisions to upgrade facilities or incentivize reuse practices.

Finally, the report emphasizes the safety and benefits of wastewater reuse, helping to dispel negative perceptions and encourage public support. Public engagement is essential for the successful implementation of water reuse programs, especially in urban areas and regions facing water scarcity.

Potential exploitation of the results

ULTIMATE is focused on water smart industrial symbioses (WSIS) between the industrial sector and services providers of the water sector. The WSIS approach is the basis for a successful implementation of circular economy technologies, because one partner produces the resource for the circular economy solution and the other partner has the demand for the recovered product. Thus, they cooperate for their mutual benefits.

The results included in this report highlight their potential for their scaling-up and exploitation in several cases of study as well as their replicability.

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Table of Contents

The project leading to this application has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 869318

The project leading to this application has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 869318

Table of tables

The project leading to this application has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 869318

The project leading to this application has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 869318

Table of figures

The project leading to this application has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 869318

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Acronyms

AGMD	Air gap membrane distillation
AnMBR	Anaerobic membrane reactor
AOP	Advanced oxidation process
CAPEX	Capital expenditure
CEB	Chemical enhanced backwash
CEC	Chemical enhanced cleaning
CED	Capacitive electrodialysis
CIP	Clean in place
COD	Chemical oxygen demand
CS	Case study
DAF	Dissolved air flotation
DMF	Dual media filter
EBCT	Empty bed contact time
EC	Electrical conductivity
ED	Electrodialysis
EWS	Early warning system
GAC	Granular activated carbon
KPI	Key performance indicator
LSI	Langelier saturation index
MBR	Membrane biological reactor
MD	Membrane distillation
MWCO	Molecular weight cut-off
N	Nitrogen
NF	Nanofiltration
OPEX	Operational expenditure
P	Phosphorus
QMRA	Quantitative microbial risk assessment
RO	Reverse osmosis

The project leading to this application has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 869318

SDI	Silt density index
SEC	Specific energy consumption
SF	Sand filter
SBP	Small Bioreactor Platform
SWMM	Storm water management model
TAN	Total ammonia nitrogen
TMP	Transmembrane pressure
TN	Total nitrogen
TOC	Total organic carbon
TRL	Technology readiness level
TSS	Total suspended solids
UF	Ultrafiltration
VAC	Value-added compounds
WR	Water recovery
WRP	Water reclamation plant
WSIS	Water smart industrial symbiosis
iWWTP	Industrial wastewater treatment plant
mWWTP	Municipal wastewater treatment plant
Z	Zeolite
ZLD	Zero liquid discharge

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1. Introduction

Wastewater can act as a reusable resource as well as a vector for energy and materials to be extracted, treated, stored, and reused. The EU-funded ULTIMATE project will operate as a catalyst for Water Smart Industrial Symbiosis (WSIS), in which water/wastewater plays a key role within a dynamic socio-economic and business oriented industrial ecosystem.

ULTIMATE is focused on water smart industrial symbioses (WSIS) between the industrial sector and services providers of the water sector. The WSIS approach is the basis for a successful implementation of circular economy technologies, because one partner produces the resource for the circular economy solution and the other partner has the demand for the recovered product. Thus, they cooperate for their mutual benefits.

ULTIMATE will demonstrate the multiple uses of municipal and industrial wastewater through nine high-level demonstrations in Europe and the south-eastern Mediterranean from the agro-food processing, beverage, heavy chemical/petrochemical, and biotech industries. It will recover, treat, and reuse industrial and municipal wastewater, derive, and exploit energy, and extract valuable materials contained in industrial wastewater, developing, and demonstrating different technologies, as it can be seen in Table 1. It will also advance innovative collaborations between businesses, water service providers, regulators, and policymakers for a more circular and socially responsible industry.

Table 1. ULTIMATE case studies and symbioses with their resources for circular economy concepts regarding water, energy, and material (WWTP: wastewater treatment plant; SME: small and medium enterprise providing water services; WRP: water reclamation plant).

Case study	Water Smart Industrial Symbioses	Resources	Closing the cycles of WATER, ENERGY, MATERIAL		
CS1 Tarragona (ES)	**Internal symbiosis within multi-industry utility**: municipal and industrial WWTP & urban WRP	Municipal wastewater and industrial wastewater from the petrochemical complex	✓		
CS2 Nieuw Prinsenland (NL)	**Internal symbiosis within cooperative**: greenhouses & water treatment facility	Drain water from greenhouses; residual and geothermal heat	✓	✓	✓

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Case study	Water Smart Industrial Symbioses	Resources	Closing the cycles of WATER, ENERGY, MATERIAL		
CS3 Rosignano (IT)	**Municipal utility, multi-industry utility & SME**: Sewer system, municipal WWTP, WRP	Municipal wastewater mixed with seawater due to an undesired intrusion of the seawater; byproducts from industry for reuse in water treatment	✓		✓
CS4 Nafplio (EL)	**Industrial utility & SME:** industrial WWTP	Wastewater from fruit processing industry	✓		✓
CS5 Lleida (ES)	**Municipal utility & multi-industry utility**: industrial WWTP & municipal WWTP	Wastewater from brewery & municipal wastewater	✓	✓	✓
CS6 Karmiel/ Shafdan (IL)	**Municipal utility & two SMEs**: two municipal WWTPs & WRP	Wastewater from olive oil production, slaughter houses and wineries & municipal wastewater		✓	✓
CS7 Tain (UK)	**Distillery, water company, & SME**: industrial WWTP	Wastewater from whiskey distillery	✓	✓	✓
CS8 Chem. Platform Roussillion (FR)	**Internal symbiosis within multi-industry utility**: industrial WWTP	Wastewater from chemical industry		✓	✓
CS9 Kalundborg (DK)	**Municipal utility & multi-industry utility**: municipal WWTP & industrial WWTP	Wastewater from pharma & biotech industry and municipal wastewater	✓	✓	✓

As it can be checked in table above, not all the nine case studies are focused in closing the cycles of water, energy, and material recovery. For this reason, different pilot plants have been designed and built to demonstrate the feasibility of different technologies, depending on the final purpose and objectives of each case study, as it can be seen in Table 2.

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Table 2. *Overview about the ULTIMATE solutions: relevant for D1.3 are the blue coloured technologies.*

Water Smart Industrial Symbiosis — Industrial Sectors: AgroFood, Beverage, Chemical/Petrochemical, BioTech; Service Providers: Municipal utility, Multi-industry utility, Specialist SME providing water services

Explanation of colour code/scale indication: WATER RECLAMATION AND REUSE; NUTRIENT & MATERIAL RECOVERY & REUSE; ENERGY & HEAT RECOVERY & REUSE; NO PILOT PLANT --> NOT PART OF D1.2; COMBINATION OF THE CS4 PILOT PLANTS FOR WATER & MATERIAL

Technologies applied & Circular Economy contributions

CS	Name			
1	Tarragona (ES)	Zeolite adsorption for ammonia removal from urban reclaimed water, reducing energy consumption of urban WWRP *TRL 5 → 6*	nZLD systems (membranes) for industrial water reuse *TRL 5 → 7*	Concept study for integration of urban and reclaimed water production for industrial water use *TRL 4 → 6*
2	Nieuw Prinsenland (NL)	Water treatment solution for recycling of drainwater from greenhouses allowing safe reuse in horticulture *TRL 4 → 6*	Closed loop greenhouses with water and nutrient recycling *TRL 4 → 6*	HT-ATES for use in greenhouse horticulture to balance out energy supply and demand using industrial residual heat *TRL 5 → 7*
3	Rosignano (IT)	Real-time data driven process control for salinity management to improve reclamation yield from municipal WWTP *TRL 5 → 7*	Data-driven matchmaking platform for water reuse of water from various sources *TRL 5 → 7*	Use of industrial byproducts as wastewater treatment process chemicals in ARETUSA reclamation plant *TRL 4 → 7*
4	Nafplio (EL)	Water reuse in industry after filtration, adsorption, super critical water extraction & AOP *TRL 5 → 7*	Mobile wastewater treatment unit for use in seasonal food processing industry combing both water recovery and material recovery units *TRL 5 → 7*	Extraction of value added compounds from fruit processing wastewater by filtration, adsorption and supercritical fluid extraction *TRL 5 → 7*
5	LLeida (ES)	Water reuse after treatement with AnMBR and ELSAR with fit-for-purpose post-treatmet: NF & RO: *TRL 7 → 9;* AOP & UV: *TRL 7 → 9;* *Online Monitoring: TRL 5 → 7*	Concept study for nutrient recovery via digestate application in agriculture TRL 5 → 7 Solar-driven hydrothermal carbonisation plant for biochar production *TRL 5 → 6*	Increased yield in biogas production in anaerobic membrane bioreactors AnMBR: *TRL 7 → 9* ELSAR: *TRL 5 → 7* and biogas valorisation: SOFC: *TRL 7 → 9*
6	Karmiel, Shafdan (IL)	Shafdan: Combined immobilised high rate anaerobic filter (AAT) with membrane filtration and activated carbon (AC) for increased biogas production *TRL 5 → 7*	Extraction of value added products from olive mill wastewater by adsorption & supercritical fluid extraction *TRL 5 → 7*	Karmiel: AAT for biogas production from poorly degradable organic matter *TRL 5 → 8*
7	Tain, Scotland (UK)	RO treatment of AnMBR effluent for water reuse in cleaning processes at the distillery *TRL 5 → 7*	Ammonia recovery from distillery wastewater *TRL 5 → 7* Struvite recovery *TRL 5 → 7*	Heat recovery from AnMBR effluent *TRL 5 → 7*
8	Saint Maurice, l'Exil (FR)	Flue gas scrubbing & dust removal for sulphur recovery as sodium bisulphite *TRL 4 → 6*	Concept study for a method to recover metals (e.g. Fe, Cu, Zn, Ni, Cr) from flue gas cleaning water *TRL 4 → 6*	Concept study to recover heat from the flue gas washing water for steam or electricity production *TRL 2 → 4*
9	Kalundborg (DK)	Combination of novel ultrafiltration membranes as pre-treatment for wastewater with high-nondegradable organic matter *TRL 5 → 7*	Concept study for nutrient and/or high-value product recovery (*Integration of solutions of other sites with TRL > 6*)	Data driven control system to increase energy efficiency through a synergetic operation of an industrial and municipal WWTP *TRL 5 → 8*

2. ULTIMATE technologies and new approaches for water recycling

This deliverable is one of three deliverables that present the results of the ULTIMATE technologies and focuses on water recovery and reuse. Deliverable 1.4 (Kleyböcker et al., 2024) and Deliverable 1.5 (González Camejo et al., 2024) deal with energy recovery and reuse and material recovery, respectively.

Table 3 provides an overview of the case studies and water recovery technologies conceptualised, developed, optimised and demonstrated in ULTIMATE.

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Table 3. Overview about the water recovery related case studies in ULTIMATE showing the resource for water recovery and the used technology.

CS	Technology	TRL	Water type produced or saved	Inflow flowrate/ capacity
1	UF+RO+MD	5→7	Cooling water	0.5-1 m^3/h (UF/RO)
1	Ammonium adsorption on zeolite	5→6	Cooling water	150-250 L/h
2	ED/(C)ED	6→7	Irrigation water	1-4 m^3/day
3	SWMM+RO	5→8	Irrigation water or industrial water, depending on the quality	450-800 L/h
4	VAC adsorption/extraction+ coagulation+AOP reactors+small bioreactor platform	6→7	Irrigation water	10 m^3/day
5	NF+RO	7→9	Cooling water	0.8-1.6 m^3/h
7	RO	5→7	Cleaning water and cooling water	0.1 L/s
9	Dynasand/media filter + UF/u-t UF/NF+RO	5→7	Industrial water	0.6-4.8 m^3/h (UF/NF) 0.2 m^3/h (RO)

The objective of this deliverable is to explain the innovative ULTIMATE water recovery solutions, to present their performance to demonstrate their feasibility and to provide recommendations for best practice implementation and application of the technologies under different process conditions.

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2.1. Increasing reclaimed water availability of the petrochemical complex of Tarragona (CS1, ES)

2.1.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

The Petrochemical Complex of Tarragona (Spain) is an industrial area that groups several companies related to the chemical and oil fields. This complex started its operation in 1971, with the construction of the first refinery, and since then its activity has progressively grown until being considered one of the most important of this type in Catalonia, Spain and southern Europe. The more than 30 companies that form this complex, from which we can highlight companies like Repsol (chemical, petroleum, and gas), Bayer, BASF, ERCROS, Cepsa, Bic or The Dow Chemical Company, are mainly focused on the production of chlorine, alkaline salts, oxygen gas, fertilisers, insecticides, fuels, plastics and synthetic essences.

Figure 1. CS1: Petrochemical complex of Tarragona (Spain).

Aguas Industriales de Tarragona Sociedad Anónima (AITASA) is a private company founded in 1965 to supply water to industries, mainly the chemical industries that were then being stablished in the Tarragona complex. AITASA supplies water for industrial and drinking uses to the complex from groundwater and reclaimed water production.

To meet its water demands in both the industry and households, Tarragona's region has traditionally relied on water transfers from the Ebro River via a system that was built back in 1989. However, the increasing water demand from the industry outpaced the system's capacity, which led to the implementation of a reclamation plant to feed industrial water only and to avoid consuming resources of the drinking water production.

Since 2012, AITASA operates the Water Reclamation Plant (WRP) of Camp de Tarragona, treating an inlet flow rate of 1200 m^3/h from two municipal wastewater treatment plants effluents, and producing 780 m^3/h of reclaimed water for boilers and cooling towers of the industry, according to the following process scheme.

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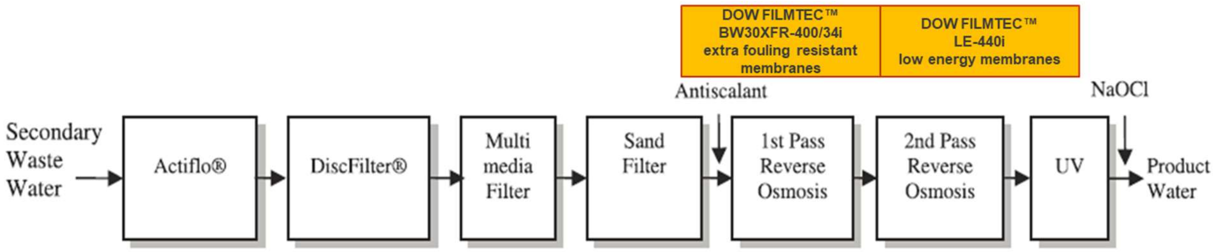

Figure 2. CS1: AITASA Water Reclamation Plant process scheme.

Reclaimed water must fulfil with Spanish Royal Decree 1620/2007 that includes the water requirements to be reused in the industry.

Table 4. CS1: Water quality requirements for reuse in cooling towers according to Spanish Royal Decree 1620/2007 (section 3.2.a.).

Parameter	Requirement	Units
Legionella	Absence	cfu/1 L
Nematode eggs	<1	eggs/10 L
Escherichia coli	Absence	cfu/100 mL
Suspended solids	<5	mg/L
Turbidity	<1	NTU

Additionally, some restrictions are established for the reclaimed water quality by the industries at the outlet of the WRP to be reused in cooling towers:

Table 5. CS1: Water quality limits for reclaimed water in Tarragona WRP.

Parameter	Requirement	Units
Ammonium	< 0.8	mg/L
Ortho-PO_4	<3	mg/L
BOD_5	<4	mg/L
TOC	< 15	mg/L
Conductivity	2000	µS/cm

This locally available additional water supply, reclaimed water, replaces surface water supplies that were transferred from the Ebro River some years ago for the use at the petrochemical park. As a result, an equivalent volume of surface water is available for urban water supply in the coastal areas of Tarragona province.

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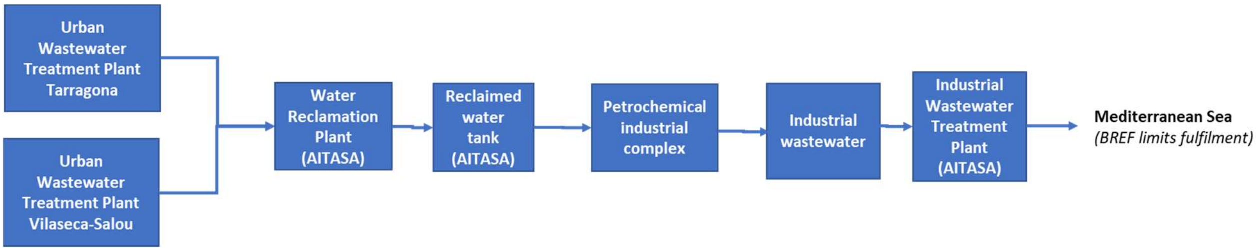

Figure 3. CS1: Scheme of the pre-existing system and the partners of the symbiosis before the start of ULTIMATE.

By developing this new and locally available water supply source, industrial growth in a water scarce region has been supported, while promoting local industry's sustainability. In April 2022, an Industrial Wastewater Treatment Plant (iWWTP) was put in operation to treat 1350 m^3/h (nominal inlet flow rate) of industrial wastewater from the different companies of the complex. In Figure 3 it can be seen the existing system to provide reclaimed water to the petrochemical complex and the iWWTP to treat industrial wastewater from this complex.

CS1 aims to extend the water synergies already implemented in the complex by increasing water availability for future. One possible water source is further treated effluent from the iWWTP to produce industrial fit-for-purpose water (cooling water).

The adsorption of ammonium with zeolites will be studied in parallel as an alternative to the second pass of the osmosis of the WRP, reducing energy consumption and increasing water recovery in the current process.

Description of the technology, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

The wastewater from the petrochemical complex is treated in an iWWTP, which includes the following processes: dissolved air flotation (DAF), membrane biological reactor (MBR) and granular activated carbon (GAC). The iWWTP effluent (1350 m^3/h) has to fulfil with Best Available Techniques Reference Documents (BREF) limits before being discharged to the environment. In Table 6, the analytical characterization of the iWWTP effluent quality is shown.

Table 6. CS1: Analytical characterization of the industrial effluent (pilot plant inlet water).

Parameter	Units	Min.	Max.	Aver.	Standard deviation	Discharge limits
pH		7.3	8.3	7.7	0.3	-
Conductivity	mS/cm	12000	24300	17140	3969	-
Suspended solids	mg/L	2.4	56	20.8	20.9	25
TDS	mg/L	8000	16000	12000	2608	-
Turbidity	NTU	0.6	11.0	3.8	4.1	-

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Parameter	Units	Min.	Max.	Aver.	Standard deviation	Discharge limits
COD	mg/L	8.5	425	114.6	171.9	100
TOC	mg/L	9	64	23	18	33
Oils and greases	mg/L	<5	<5	<5	2,9	-
Ammonium	mg/L	0.1	8.3	3.5	3.4	-
TIC	mg/L	69.8	98.6	79.7	10.3	-
Carbonate	mg/L	<6	<6	<6	0	-
Bicarbonate	mg/L	330	450	396.7	40.8	-
Chloride	mg/L	2400	5930	4626	1212	-
Bromide	mg/L	2.4	31	17.2	13.6	-
Sulfate	mg/L	1100	2000	1491	263	-
Total P	mg/L	1	3.5	1.9	0.9	3
Phosphate	mg/L	2.5	10	4.5	3.2	-
Total N	mg/L	3.3	22	10.2	6.7	25
Nitrite	mg/L	0.1	4.4	2.6	1.8	-
Nitrate	mg/L	4	43	20.9	14.9	-
Calcium	mg/L	170	230	204	97	-
Magnesium	mg/L	43	110	63.8	22.1	-
Barium	mg/L	0.1	0.1	0.1	0	-
Sodium	mg/L	3200	4720	3645	2014	-
Potassium	mg/L	26	58	38.8	10.5	-
E.Coli	nmp/100 mL	0	7	2.8	2.9	-

According to the values collected in Table 6, iWWTP effluent has a high variability in quality, mainly in terms of conductivity, COD and suspended solids, and furthermore, it has a scaling tendency according Langelier Saturation Index (LSI>0).

An nZLD system based on membrane technologies is proposed for the treatment of industrial wastewater treatment plant (iWWTP) effluent. As shown in Figure 4, the effluent first undergoes an ultrafiltration (UF) process; the resulting permeate is then treated in a reverse osmosis (RO) system, and the RO concentrate is processed in a membrane distillation (MD) unit.

On the other hand, ammonium adsorption on zeolites is proposed as lower energy consumption technology to replace the reverse osmosis second pass in the Water Reclamation Plant and increase water recovery of the global process.

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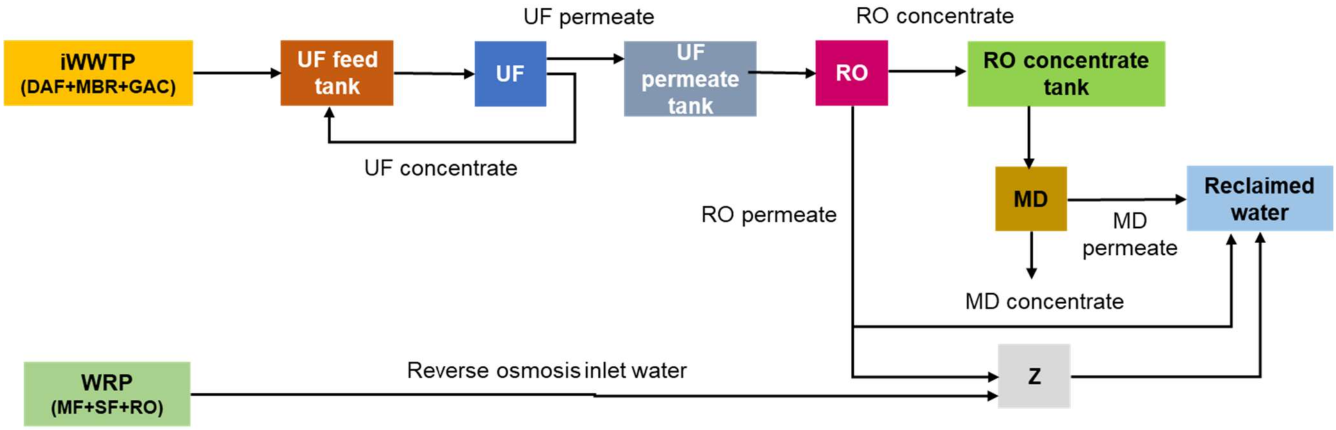

Figure 4. CS1: Pilot plant process scheme.

The UF system consisted of two 8 mm tubular polymeric membranes Memos ME-C100-08-2995-6.2 (2995 mm length), with 6.2 m^2 of surface area each. The UF system was operated with recirculation. Whereas the permeate was collected in a 5m^3 tank, the concentrate was recirculated back to the 10m^3 feed tank where it was mixed with feed water. The reason to use recirculation was to increase the amount of shear acting on the membrane surface which will lessen fouling caused by extreme concentration polarization when treating concentrated feeds. Due to the recirculation of the concentrate, a suspended solids concentration effect could be observed in the UF feed tank, and for this reason, this tank was purged every 2 weeks.

A typical process cycle consisted of a production period and a flushing period. During the production period, permeate was produced by the UF system. During the flushing period, permeate was pumped through the membrane to scour particles that were on the membrane surface.

In RO unit, there are two low fouling spiral wound membranes Hydranautics SWC5-LD (seawater). The RO system starts from the UF permeated water reservoir and is pumped through a 5 µm cartridge microfiltration to protect the modules and extend the intervals between CIP cleanings of the membranes. Once the water passes through the filter, it reaches the feed pump at a pressure of approximately 1 bar, raising the pressure to the level required by the RO (around 22 bar). This plant is designed with an energy recovery system that takes the permeate stream at a slightly lower pressure than the feed and raises it by 1 or 2 bar, generating a sweeping flow on the feed side of the membrane. The feed flow rate can be adjusted, which not only saves energy but also allows operation at different feed flow rates, something we consider convenient for the project's research personnel.

Like the UF, the RO requires a rinse in case of system shutdown and periodic CIP cleanings in a closed circuit. The RO system will have two dosing systems to condition the feed water: one for antiscalant to prevent inorganic fouling of the RO, and another pump to dose either acid or base to adjust the pH according to the conditions of each experiment.

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The MD pilot is designed to operate in a batch mode. The IBC container which is filled with the feed water can be operated until a certain concentration is reached. Then the unit will stop automatically and needs to be flushed with clean water or permeate.

In *Table 7* the membrane specifications are listed.

Table 7. CS1: Pilot plant membrane characteristics summary.

Parameter	Ultrafiltration	Reverse Osmosis	Membrane distillation
Supplier	Memos	Nitto, Hydranautics	Gore
Membrane reference	ME-C100-08-2995-6.2	SWC5-LD	
Material	Polyvinylidene fluoride (PVDF)	Polyamide	Polytetrafluoroethylene (PTFE)
Surface	6.2 m^2	37.2 m^2	11 m^2
Pore size (cut-off)	100 kDa	n.a.	0.2 μm
Flux	>300 L/(m^2h100 kPa)	34.1 m^3/d (5.5 MPa, 10% recovery)	
Transmembrane pressure	-40 to 800 kPa (40ºC)	0.10 MPa	
Crossflow velocity	2 to 5 m/s		
Max. temperature	40ºC (800 kPa)	45ºC	

For adsorption tests, natural zeolite, ZN Aqua 0.5-1mm (82-86% clinoptilolite) by Zeocat was used in a 140 L column. Water from WRP (sand filters outlet according Figure 2) was fed downstream and, once the zeolite was saturated (at breakthrough point), it was used 10% NaCl solution for regeneration.

The four technologies were installed in a 40 feet maritime container at AITASA facilities and tested independently, working in discontinuous mode.

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Figure 5. CS1: Pilot plant technologies: ultrafiltration (top, left), reverse osmosis (top, right), membrane distillation (bottom, left), zeolite column (bottom, right).

UF tubular membranes installed can be backwashed with water or chemicals against the filtration direction. Also, chlorine can be used as cleaning agent. For the UF tests, no coagulant was dosed, because inlet water comes from a previous MBR process with UF membranes in the iWWTP.

For the RO spiral wound membranes, the feed water must be free from particles (NTU<1). In case of fouling or scaling a clean in place (CIP) can be carried out with acid and caustic, chlorine is not possible. The pre-treatment membranes protect the RO from organic and/or colloid fouling, retain particles and bigger molecules. The retained impurities can be removed easier by backwash and chemical cleaning compared to RO.

The TRL of the operation of the near zero liquid discharge (nZLD) membrane system (UF+RO+MD) increased from 5 to 7. The capacity of the technology can be easily adapted and expanded to the needs of AITASA. The technology can directly be used for the treatment of the iWWTP effluent of Camp de Tarragona and can also be transferred to other industrial sites. Here the legal basics have to be checked in advance (mainly, discharge regulations).

The TRL of the zeolite adsorption increased from 5 to 6. This technology is not as easy to implement as the nZLD system, as it would replace an already existing industrial facility. AITASA shifted their preliminary interest to studying this technology as a parallel process to the current second step of the RO in the Water Reclamation Plant.

Technical requirements for its implementation and operating conditions

Table 8 shows the feed water requirements for the application of UF/RO membranes. If the chemical composition of the WWTP effluent does not comply with the indicated ranges, a pre-treatment prior to the inflow to the unit is recommended, e.g. sand filtration or multimedia filtration.

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Table 8. CS1: Typical feed water conditions to apply UF and RO.

Parameter	Units	Ultrafiltration	Reverse osmosis
Temperature	°C	<70	<45
pH	-	2-10	2-11
Turbidity	NTU	n.a.	<1
SDI (15 min)	-	n.a.	<5
Free chlorine	mg/L	<250000 ppm h	<0.1

In the following table, operational conditions for the membrane technologies are listed.

Table 9. CS1: Tested operational conditions in ultrafiltration and reverse osmosis.

Parameter	Unit	Ultrafiltration	Reverse osmosis
Inlet flow rate	m^3/h	22-49	0.8-1
Inlet pressure	bar	0.3-3	16-56
Flux	$L/(m^2 h)$	8-242	2.3-10.9
Crossflow velocity	m/s	1.5-3.4	-

For UF, different frequency of backwash and clean in place (CIP) procedures were tested.

Table 10. CS1: Tested operational conditions in membrane distillation.

Parameter	Unit	Membrane distillation
Inlet flow rate	L/h	378-542
Inlet temperature	ºC	68-80
ΔT	ºC	30-50
Inlet pH	-	7.5-8.8
Flux	$L/(m^2 h)$	2.3-4.4

Table 11. CS1: Tested operational conditions in zeolite adsorption.

Parameter	Unit	Zeolite
Inlet flow rate	L/h	150-250
Ammonium inlet	mg/L	30-65
Empty bed contact time (EBCT)	min	6.8-40

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2.1.2. Results of new approaches

Results of feasibility study and expected technology performance (KPIs)

Ultrafiltration

Pilot plant is fed with the outlet stream from the Industrial Wastewater Treatment Plant. According to the analytical results in Table 6, there are suspended solids in the effluent, and for this reason, ultrafiltration has been installed to protect reverse osmosis.

An inlet mesh filter (2 mm) was installed before UF inlet and cleaned when pressure drop reached 1 bar. Despite the suspended solids in feed water, and its scaling tendency (Langelier Saturation Index LSI=0.7-1.3), any coagulant and antiscalant reagent were added at the ultrafiltration inlet.

The performance of the UF process was mainly monitored over flux values and transmembrane pressures. Due to the high variability of the quality of feed water (see Table 6), great variations in permeability and transmembrane pressure (TMP) was observed.

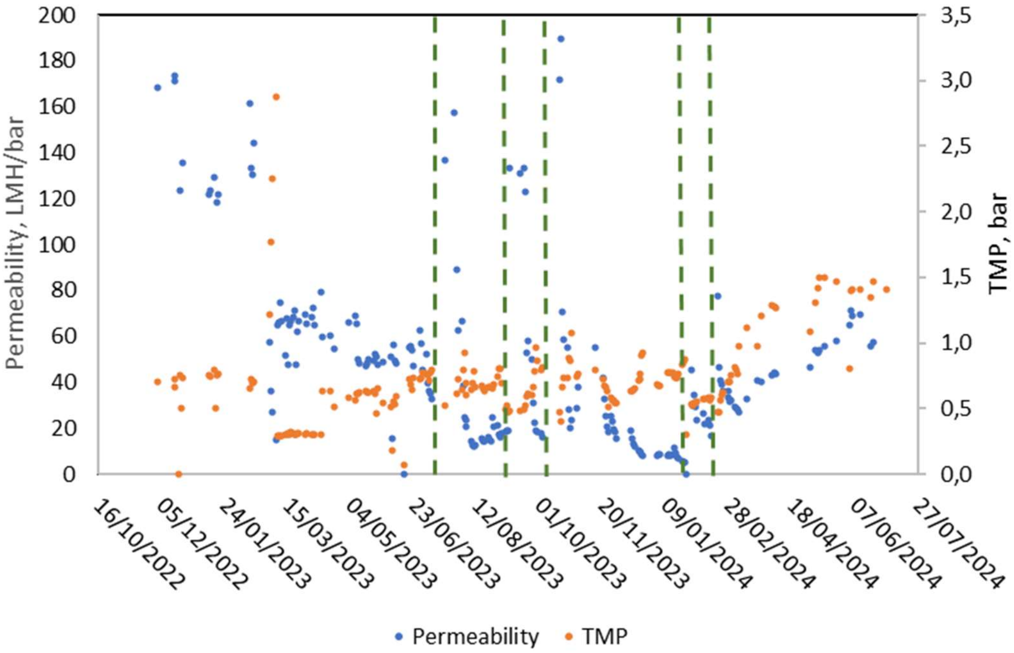

Figure 6. CS1: Ultrafiltration permeability and transmembrane pressure (max. 8 bar). Green dotted lines show CIP cleanings.

As it is collected in Table 9, inlet flow rate was in the range of 22-49 m^3/h, with a crossflow in the range 1.5-3.4 m/s (supplier recommendation is 2-5 m/s). CIP cleanings were conducted when an increase of TMP or decrease in permeability were observed using 400 ppm NaOH and 2000 ppm NaClO solutions. As it can be observed in Figure 6, permeability increases significatively after CIP cleanings.

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Ultrafiltration decreased 86% the turbidity. The content of TSS and COD decreased as well, 51% and 18% respectively (average).

Regarding specific energy consumption (SEC), it was calculated theoretically, obtaining an average value of 0.7 kWh/m^3.

Reverse osmosis performance

RO process was fed with UF permeate. The performance of the RO process was monitored over permeability values and transmembrane pressure. Throughout the entire experimentation, the inlet flow rate was fixed at 1 m³/h, and recovery ratio was modified to assess its impact on the system performance, starting with 45% recovery until 80%. The only chemical agent that was dosed throughout the entire experimentation was antiscalant Hydrex 4106 (by Veolia), 4 mg/L dose.

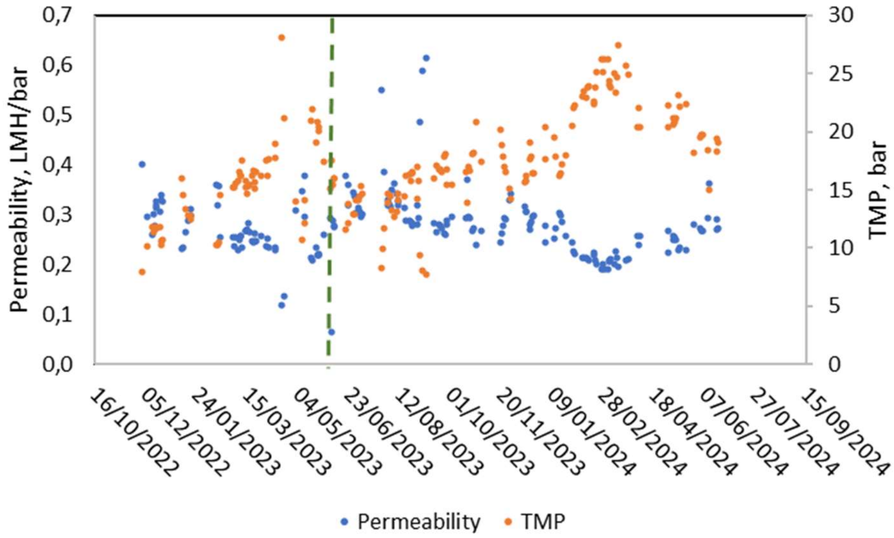

Figure 7. CS1: Reverse osmosis permeability and transmembrane pressure. Green dotted line shows CIP cleaning.

An average permeability of 0.3 LMH/bar was achieved during the experimentation period. During RO operation, a basic and acid CIP cleaning was conducted in June 2023 to increase permeability (33%) and decrease transmembrane pressure (27%).

Regarding RO permeate quality, conductivity and ammonium concentrations are the parameters of most concern, and for this, they were measured periodically (see Table 13). Ammonium concentration in permeate is lower than required limit, 0.8 mg/L to be used as reclaimed water in petrochemical facilities. However, conductivity exceeds established limit, 40 µS/cm, and for this reason, a second pass RO was tested, entering RO permeate in RO process again. Recovery between 67% and 85% was tested, and rejections were between 97% and 98%, while the conductivity was always below 4 µS/cm. 2-pass RO permeate analytical quality can be checked in Table 13.

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Membrane distillation

Reverse osmosis concentrate was treated with Air Gap Membrane distillation (AGMD), with 378-542 L/h inlet flow rate. Feed temperature varied between 69 and 80ºC, with a temperature gap between 30 and 50ºC. Any kind of pH adjustment of feed was previously made.

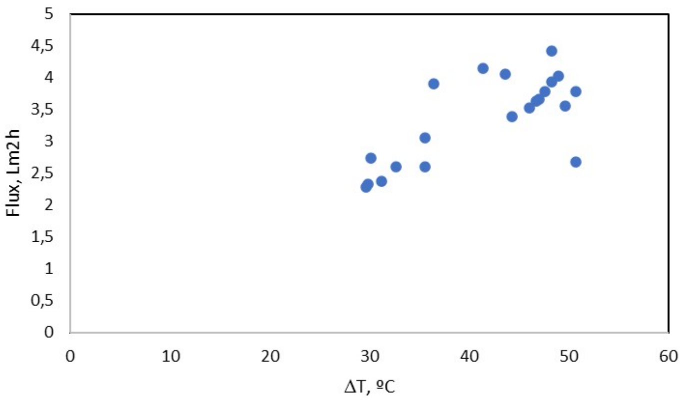

Figure 8. CS1: Membrane distillation permeability.

In terms of permeate quality, the permeate did not meet required limits, mainly in terms of conductivity and ammonium (see Table 13). According to Davey C.J et al. (2021), the ratio of ammonium to ammonia in solution is a function of the solution pH and temperature and can therefore be predicted from the dissociation constants according to the equilibrium NH3 + H2O⇌NH+ 4 + OH−. Considering that membrane distillation feed pH is 8 (aver.) and temperature is 75ºC (aver.), it is estimated that 50% of the ammonium is in solution in the feed. For this reason, initial pH adjustment in feed is required, adding acid, to avoid ammonium passing the permeate.

nZLD system

In this section, a summary is provided about operational parameters and permeate quality.

Table 12. CS1: Tested operational conditions in ultrafiltration and reverse osmosis.

Parameter	Unit	Ultrafiltration	Reverse osmosis	Membrane distillation
Inlet flow rate	m^3/h	22-49	0.8-1	0.4-0.5
Inlet pressure	bar	0.3-3	16-56	0.3-0.5
Flux	L/(m² h)	8-242	2.3-10.9	2.3-4.4
Permeability	LMH/bar	5-190	0.1-0.6	7.2-11.9
Recovery	%	20-90	45-82	7.8-10.8

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Parameter	Unit	Ultrafiltration	Reverse osmosis	Membrane distillation
SEC (aver.)	kWh/m^3	0.7	1	n.a.

Regarding product quality, in Table 13 obtained permeate is compared with reclaimed water required limits. As it can be checked, only 2-pass RO permeate met reclaimed water quality requirements, while membrane distillation permeate does not at the tested operation conditions.

Table 13. CS1: Comparison of obtained water quality (average values) and reclaimed water limits.

Parameter	Unit	Reclaimed water limits	UF permeate	1-pass RO permeate	2-pass RO permeate	MD permeate
Conductivity		20-40	12274±3938	154±185	<10	281±420
Ammonium	mg/L	0.8	1.7±3.2	0.2±0.2	0.1	1.4±1.4
Suspended solids	mg/L	5	4.6±4.2	0.3±1.2	<2	0
Turbidity	NTU	1	0.4±1	0.4±1.3	0.5	0.4±0.2
TOC (COD)	mg/L	15	48±21	9.8±6.3	1.1	14.8±8.2
E. Coli	nmp/100 mL	0	n.a	n.a	n.a	n.a

Global recovery of the ultrafiltration and 2-pass reverse osmosis system is depicted in Figure 9. As it can be checked, ultrafiltration recovery has a great impact on the global one. Global recovery KPI was set in 20-30%, and according to the obtained results, this recovery can be achieved in UF+2-pass RO system when ultrafiltration is operated properly. If it is considered a recovery ratio of 70% for the first pass of the RO and 85% for the second pass, recovery higher than 40% in UF is required to meet a global recovery higher than 20%. 40% recovery in UF can be easily achieved if the process is operated properly.

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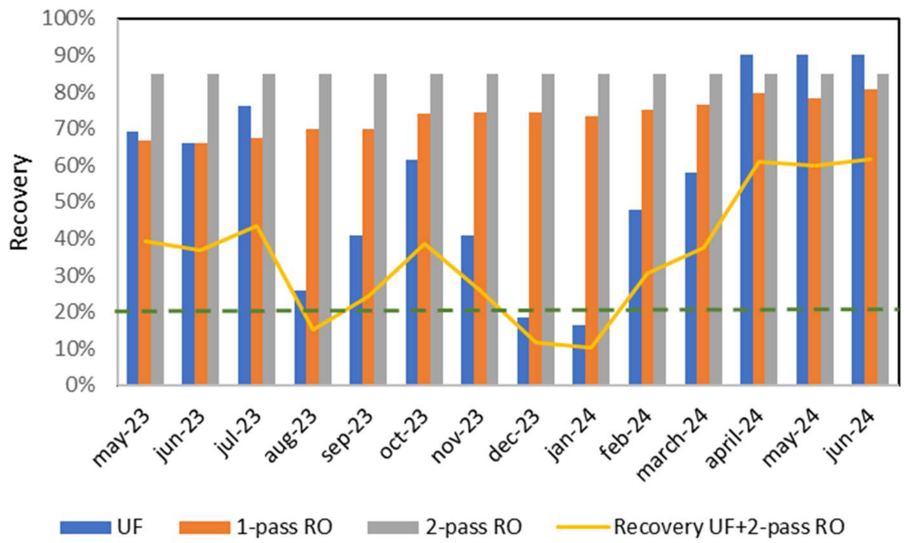

Figure 9. CS1: Ultrafiltration and 2-pass reverse osmosis global recovery.

On the other hand, in case of scaling up these technologies, it should be considered that concentrate effluents would be discharged, having to fulfil BREF limits.

Table 14. CS1: Comparison of effluents to be discharged (average values) and current discharge limits.

Parameter	Unit	Discharge limits	UF concentrate	1-pass RO concentrate	MD concentrate
TOC	mg/L	33	21.3	32	48
COD	mg/L	100	231	663±703	n.a
Suspended solids	mg/L	25	6.3	5.2±5.9	0
Total N	mg/L	25	10	18.3±8.4	n.a
Total P	mg/L	3	1.3	9.4±6.6	n.a
Chromium	µg/L	25	n.a	<2	n.a
Copper	µg/L	50	n.a	2.1	11
Nickel	µg/L	50	n.a	21	30
Zinc	µg/L	300	n.a	220	n.a
Cadmium	µg/L	8	n.a	<0.2	n.a
Mercury	µg/L	1	n.a	<1	n.a
Lead	µg/L	30	n.a	<1	n.a

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According to results in Table 14, UF, RO and MD concentrate streams do not fulfil the discharge limits, and for this reason, a post-treatment could be necessary in case these streams are discharged into the sea.

Finally, given the industrial origin of the treated water, the concentrations of critical raw materials (CRM) in the reverse osmosis and membrane distillation concentrates were analyzed. The metals found in the highest concentrations were strontium, boron, zinc and lithium.

Table 15. CS1: Critical Raw Materials (CRM) in reverse osmosis and membrane distillation concentrates (the five most concentrated).

CRM	Units	Reverse Osmosis	Membrane distillation
Strontium	mg/L	5671	14600
Boron	mg/L	469	860
Zinc	mg/L	220	
Lithium	mg/L	105	269
Manganese	mg/L	52	35
Nickel	mg/L		30

Ammonium adsorption on zeolites

Adsorption trials with zeolite were conducted with water from the Water Reclamation Plant, after sand filters process (Figure 2) to treat water with higher ammonium concentration (30-65 mg/L) than in reverse osmosis permeate (0.02-1 mg/L).

Saturation curves were obtained to determine break-through time (0.8 mg ammonium/L at the outlet) and determine zeolite ammonium adsorption capacity. Trials were carried out comparing two empty bed contact time (EBCT) of 6.8 min and 40 min.

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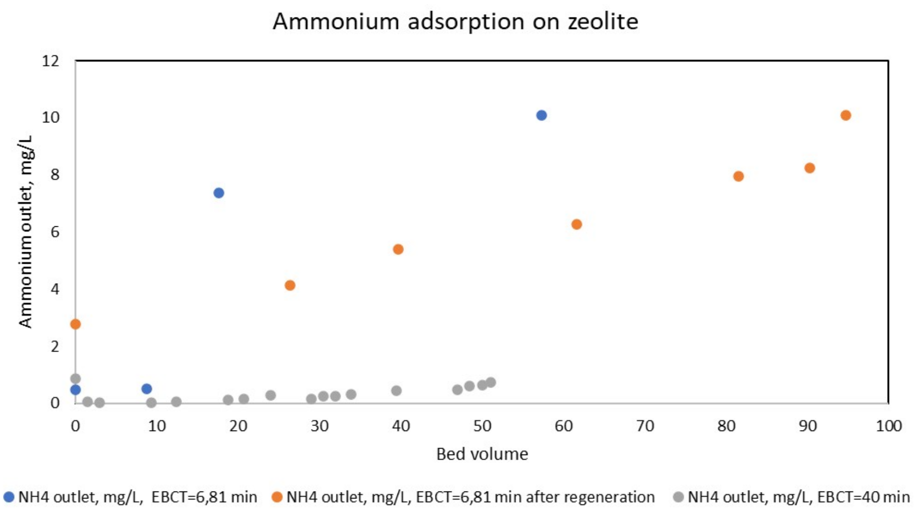

Figure 10. CS1: Ammonium adsorption on zeolites.

Breakthrough time for the first experiment was 1 h, and the ammonium adsorption capacity was 0.7 mg ammonium/g zeolite, lower than values in literature (Guida et al. 2021). After regeneration with a NaCl 10% solution, a second trial was conducted, exceeding the ammonium limit during the first minutes of the experiment, and for this reason breakthrough time is not available. A third trial was carried out, with new zeolite and increasing contact time until 40 min. In this last trial, ammonium adsorption capacity was 2.5 mg ammonium/g zeolite.

Table 16. CS1: Adsorption operational conditions and adsorption capacity.

Trial	Units	1	2	3
Zeolite mass	kg	14.3	14.3	140
Observations (zeolite)	-	New	Regenerated	New
NH4 inlet, mg/L (average)	mg/L	64.7	30.4	41.3
Inlet flow rate	L/h	150	150	250
Linear velocity	m/h	5.5	5.5	2.3
Empty bed contact time (EBCT)	min	6.8	6.8	40
Breakthrough time	h	1	-	34
Adsorption capacity	mg NH_4/g zeolite	0.7	-	2.5

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Water quality at the adsorption column outlet can be seen in Table 17.

Table 17. CS1: Comparison of water quality at the inlet and outlet of the adsorption column at break-through point (EBCT=6.8 min). Tests conducted with water from Water Reclamation Plant, sand filters outlet water.

Parameter	Unit	Inlet(*)	Outlet	Removal, %
pH	-	6.7	6.8	-
Conductivity	µS/cm	2800	2740	2.2
Turbidity	NTU	1.6	3.8	-76
TSS	mg/L	21	8	62
COD	mg/L	30.6	23.5	23
Ammonium	mg/L	64.7	0.5	99

According to the results in Table 17, TSS and COD concentration are reduced, 62% and 23% respectively. Turbidity increases 76%, with the main hypothesis being that the water carries the zeolite itself. However, conductivity is barely reduced, only 2.2.%, This result means that ammonium adsorption technology is only feasible in case inlet water conductivity < 40 µS/cm.

Considering operation conditions and adsorption capacity obtained in trial 3, a LCA was developed and explained in D2.2, comparing the feasibility of replacing the current two-step reverse osmosis (RO) scheme (Figure 2) with a system where the second RO step is substituted by a zeolite adsorption system. This new approach by ULTIMATE proposes incorporating a system that uses fewer resources by optimizing energy consumption and eliminating the use of sodium hypochlorite. This will enable the production of reclaimed water of a quality suitable for reuse within the same industrial complex. Regarding the results from LCA analysis, 6% energy consumption savings (KPI:10%) can be achieved replacing the reverse osmosis second-pass with a zeolite adsorption system by gravity.

Comparison of baseline situation with ULTIMATE solution

For the case study, information was initially collected to quantify and assess the baseline conditions and address petrochemical complex companies concerns. Additionally, information was gathered from site operators and facilities, concluding that, because of the current water scarcity, and alternative water source for reuse it is required.

In the current situation, the effluent from the iWWTP (1350 m^3/h) at site is discharged and not reused, resulting in a 100% loss. In contrast, implementing full-scale recovery from the effluent discharge based in the proposed scheme (ultrafiltration and 2-pass reverse osmosis) should achieve a recovery rate higher than 20-30%, depending mainly on UF performance, with the required reclaimed water quality. It means that

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more than 270 m^3/h can be produced with this technologies train. However, this solution would require high investment and additional activities to study the management of waste streams generated (concentrate).

2.1.3. Conclusion

Lessons learned from the feasibility study (technical risks & measures)

The current effluent from the IWWTP can be treated using ultrafiltration and a two-pass reverse osmosis system to produce water of sufficient quality for reuse in industrial cooling processes.

While the water entering the UF+2-pass RO system is already pretreated, ultrafiltration has proven essential for protecting the reverse osmosis stage. In fact, ultrafiltration has emerged as the critical factor determining the overall recovery rate of the system. Some of the challenges encountered in the ultrafiltration process stem from the significant variability in feedwater quality and its tendency to cause scaling. This highlights the need to add a coagulant and antiscalant to improve the efficiency of the process.

Reverse osmosis tests have been performed using seawater membranes to ensure effective contaminant rejection and achieve the required conductivity in the permeate.

Membrane distillation is an innovative technology that offers the potential to treat the concentrate from reverse osmosis and further increase recovery rates. However, due to the composition of the feed stream, additional tests are necessary, including pH and temperature adjustments at the inlet stream, to determine whether the permeate can meet the quality standards needed for reuse.

Additionally, it is important to consider that if the aforementioned technologies are scaled up, any discharged waste streams must comply with current legal discharge limits. This could become a critical factor when implementing the solution, as some parameters have been shown to fall short of these limits.

Given the industrial origin of the treated water, the concentrations of critical raw materials (CRM) in the reverse osmosis and membrane distillation concentrates were analyzed. The metals found in the highest concentrations were strontium, boron, zinc and lithium.

Lastly, natural zeolite has been shown to adsorb ammonium, though its adsorption capacity is limited, and it is not effective in reducing conductivity or retaining other contaminants. Therefore, zeolite adsorption could only serve as an alternative to the second reverse osmosis stage in the WRP if the treated stream (the permeate from the first stage) already complies with all other required parameters for reclaimed water.

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Best practices and recommendations for technology implementation in a symbiotic frame

Initially, a comprehensive characterization of the water quality to be treated is required, along with a study of the variability of the main analytical parameters and the investigation of a possible pretreatment.

On the other hand, more pilot plant-scale tests would be necessary to ensure proper scaling up. At this point, the involvement and commitment of the end-users (companies in the petrochemical complex) is essential to achieve this goal. The environmental benefits of water reuse are clear, but the economic benefits may not be, due to the low water prices in dry countries like Spain, and for this reason, companies support will be crucial. For this reason, financial incentives, such as subsidies and low-interest loans for large-scale demonstrations, are crucial for encouraging the adoption of these technologies.

Finally, public incentives or private strategies for water reuse or drought scenarios could be key drivers. Furthermore, environmental policies play an important role in this case study because, in case of scaling up the proposed technologies, new discharged streams won´t fulfil with current discharge limits, and for this reason, authorities have to assess how to face up this future situation.

To sum up, by addressing these financial, regulatory, and collaborative aspects, the adoption of the proposed technologies can lead to sustainable and efficient water reclamation for the petrochemical industry in Camp de Tarragona.

Crucial factors for technology implementation and its optimal performance

- Successful implementation for water reclamation from iWWTP showcased with the case study. Higher inlet flow rate demonstration could be useful to optimize operational conditions and obtain more accurate operational and investemet costs estimation.
- Ultrafiltration has proven to be the most critical process in the UF+2-pass RO system and defines the system's overall recovery. For this reason, UF operation must be properly studied, optimizing operating parameters as well as cleaning frequencies and protocols
- Ammonium adsorption on zeolites seems not to be an alternative for the 2-pass RO, but it could be a complementary process to decrease ammonium concentration at the RO inlet, mainly, in season period. However, it should be considered the high footprint required by this technology.
- Discharge management (concentrate) is still a technical and regulatory) issue

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2.2. Optimising water reclamation from agro-food industries in N. Prinsenland (CS2, NL,)

2.2.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

Coöperatieve Tuinbouw Water Zuivering de Vlot is a wastewater treatment facility located at 's- Gravenzande treating 160 hectares (60 companies) of wastewater from greenhouses (40-60 m^3/h) mainly growing ornamental and vegetable crops. In the coming years, this sector is faced with water availability (need for alternative high-quality sources) and discharge regulation challenges (removal of PPP by 2021 and limitations in nutrient discharge by 2027). Thus, for this facility, De Vlot aims to explore water and nutrient reuse opportunities from their wastewater (approx. 10% of the total water input) by optimizing their system for internal symbiosis within their own facility and external symbiosis with neighbouring greenhouses and industries (supported by extensive survey conducted in 2021), to reach (nearly) zero liquid discharge.

Figure 11. CS2: Greenhouse Horticulture in the Netherlands (left) and Wastewater Treatment Plant at De Vlot (right).

In ULTIMATE, water reclamation from De Vlot will be extended as follows: The wastewater stream from the greenhouse is currently discharged to the sewer, although it contains value for the water itself (0.6 €/m³). Re-use of this water to irrigation is hampered by the risk of introducing salinity (specifically Na+<0.1 mmol/l) and plant diseases. ULTIMATE will improve and demonstrate the functionality of advanced wastewater treatment for reliable removal of salinity via electrochemical methods, to produce fit-for-purpose irrigation water (Figure 12).

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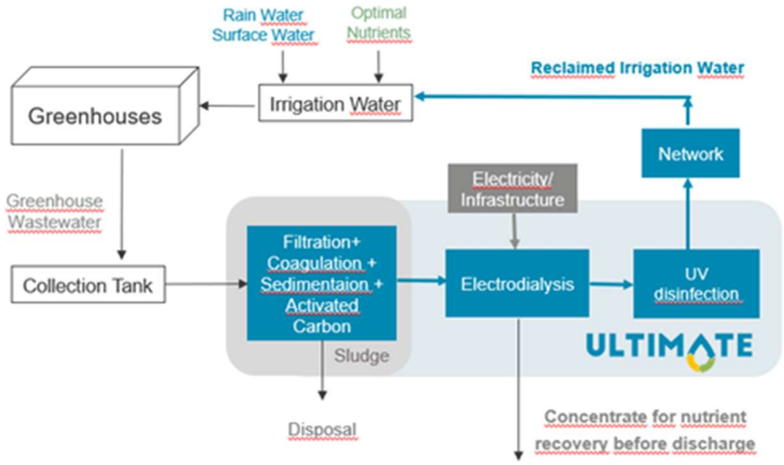

Figure 12. CS2: Treatment scheme and proposed Ultimate technology solution for the wastewater treatment plant at De Vlot.

Description of the concept, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

For electrodialysis (ED) and its process variations, the treatment potential can range from below 100 m^3/day to 20,000 m^3/day (Strathmann H. et al., 2010). The two main ED geometric patterns include sheet flow and the tortuous path, and it can operate in batch or continuous mode (Gurreri et al. 2020). Table 18 provides the range of typical (C)ED operating parameters.

Table 18. CS2: Typical range for operating parameters for (C)ED.

Parameter	Units	Range	Reference
Cell pairs	no.	<10 (lab scale) – 100s (pilot/full)	(Gurreri et al. 2020)
Active area per membrane	m^2	0.01 – 0.06 (up to 1)	(Gurreri et al. 2020)
Spacer thickness	mm	0.3 - 2	(Campione et al., 2020)
Flow velocity	cm/s	1-10 (up to 50)	(Campione et al., 2020)
Applied voltage	V	7 (lab scale) – 30 (pilot/full)	(Strathmann H. et al., 2010)
Current density	mA/cm^2	5 - 60	(Mohammadi et al. 2021)
Running time	h	2 (lab scale)-months (pilot)	(Strathmann H. et al., 2010)

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Technical requirements for its implementation and operating conditions

Table 19 gives the ED and CED operating conditions applied for CS2. For the application of (C)ED to treat greenhouse wastewater, the requirements to be met include achieving high quality irrigation water quality (Table 20), optimizing operating conditions (applied voltage, water recovery, flow velocity etc.), achieving efficient specific energy consumption, and competitive price per m^3 for the reclaimed water (as compared to other currently accepted technologies such as RO).

Table 19. CS2: ED and (C)ED technology specification.

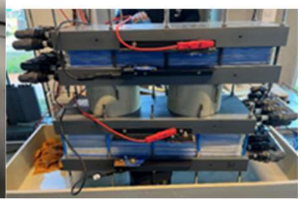
Electrodialysis Set-up	Capacitive Electrodialysis Pilot
PC Cell ED stack	Fujifilm Pilot
10 – 20 cell pairs (scalable)	150 cell pairs (Industry standard)
Membrane area 0.01 m^2	Membrane area 19.32 m^2 (Capacity 1-4 m^3/day)
Fujifilm Type 10	Fujifilm Type 10 IEMs
Batch mode	One-pass mode
PC Cell stack	150 cell pairs (Industry standard)
	 

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Table 20. CS2: De Vlot wastewater feed water composition (above). Conductivity (EC), Na+, and Cl- guidelines for greenhouse horticulture irrigation water (below) [Guleria et al., 2024].

Parameter	Abb.	EC	pH	NH_4^+	K^+	Na^+	Ca^{2+}	Mg^{2+}	NO_3^-	Cl^-	SO_4^{2-}	HCO_3^-	PO_4^{3-}
Units		*mS/cm*		*mmol/L*									
Real collective greenhouse wastewater	RCGW	1.9	8.2	0	2.3	8.41	3.38	1.74	5.00	9.10	0.20	6.23	1.36

S. no.	EC (mS/cm at 25 °C)	Na^+ (mmol/l)	Na^+ (mg/l)	Cl^- (mmol/l)	Cl^- (mg/l)
1	<0.2	<0.1	<2.3	<0.1	<3.5
2	<0.5	<1.5	<35	<1.5	<53
3	0.5–1	1.5–3	35–69	1.5–3	53–107
4	1–1.5	3–4.5	69–104	3–4.5	107–160

2.2.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

Reclaimed water quantity

For the case study, a comprehensive survey was conducted to quantify and assess the baseline conditions and address growers' concerns about reusing treated wastewater. Additionally, information was gathered from site operators. Based on these data, it is estimated that up to 12-25% of the total freshwater use can be reclaimed. This estimation is based on the average annual irrigation water requirement, which ranges from 500,000 to 800,000 m³ (for an 80-160 ha cultivation area), excluding internal recycling at the individual greenhouse level. The average wastewater influent for treatment was estimated to be 150,000 m³. With the proposed (C)ED technology, which has a water recovery efficiency of 60-90%, the estimated reclaimed water is 16% of the total freshwater use for the greenhouse consortium. This translates to an average water recovery of 150,000 m³ per year, highlighting the potential for significant water reclamation through the treatment of greenhouse wastewater

Reclaimed water quality and (C)ED performance

The composition of the generated reclaimed water (diluate) was assessed with both (C)ED technologies, as it is important, especially for fit-for-use applications such as greenhouse horticulture, to reduced certain ions to specific levels. All compositions reached the high-quality greenhouse irrigation water (Table 20). Below are the detailed results.

Electrodialysis (ED) set-up

An overview of the experimental conditions to determine the performance of the ED set-up is given in Annex 1. Figure 13 shows the conductivity removal and ionic water

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quality of the irrigation water for reuse (diluate) with the baseline conditions for reaching the target conductivities (EC in the range of 1-<0.2 mS/cm).

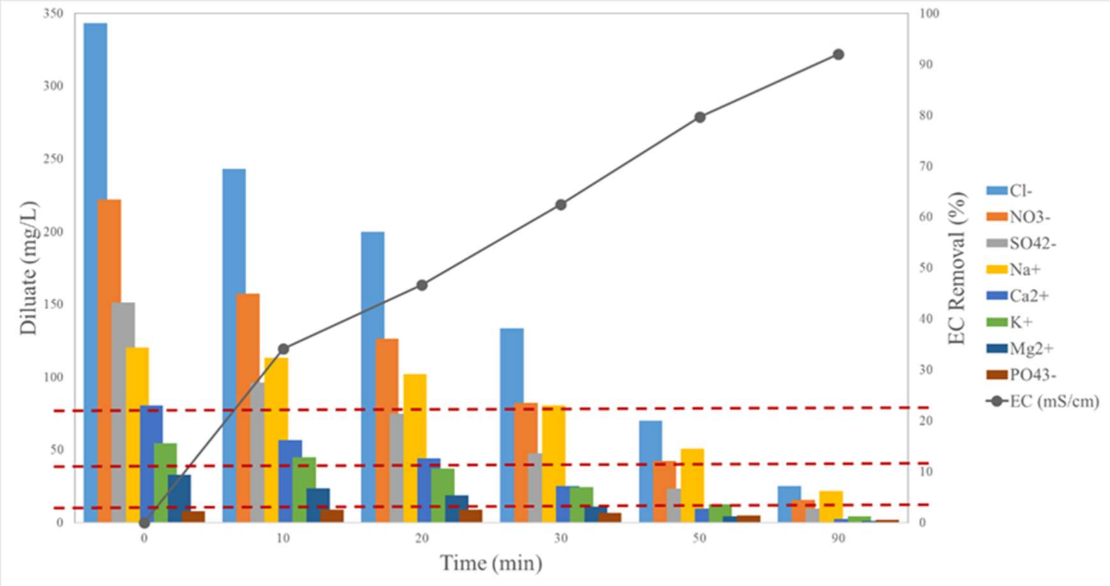

Figure 13. CS2: EC removal and ionic water quality of the irrigation water for reuse (diluate) with the baseline conditions for reaching the target conductivities (EC in the range of 1, 0.5 and <0.2 mS/cm, represented by the red dotted lines).

Figure 13 demonstrates the effectiveness of electrodialysis (ED) in removing various ions and reducing the electrical conductivity (EC) of the diluate over time. Monovalent ions such as chloride and nitrate exhibit high removal rates, indicating their high flux and permeability through the ED system. Sodium and potassium also show consistent removal, while divalent ions like sulfate, calcium, and magnesium, along with trivalent phosphate, demonstrate slower reduction due to their larger size and higher charge. The overall EC removal reaches approximately 90%, highlighting the ED system's efficiency. However, divalent and trivalent ions show lower selectivity. These results underscore the ED system's potential for significantly improving water quality for irrigation purposes, emphasizing its effectiveness for monovalent ion removal while indicating areas for further optimization in handling more complex ions.

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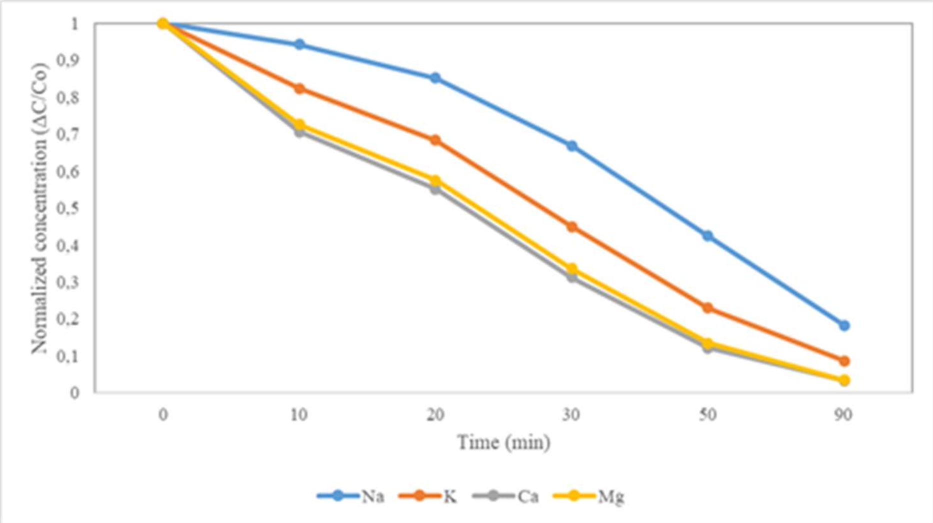

EC Removal (%)	Na/K	Na/Ca	Na/Mg
34,2	0,7	0,2	0,7
46,7	1,0	0,4	1,2
62,5	1,3	0,7	1,8
79,6	1,6	0,9	2,4
91,9	1,9	1,2	3,1

Figure 14. CS2: Normalized cation removal over time (above) and cation selectivity over EC removal (below).

Sodium (Na^+) shows the highest removal rate, with its normalized concentration decreasing significantly over time, reflecting its high mobility and permeability in the ED system. Potassium (K) also exhibits a steady decrease, though at a slightly slower rate compared to sodium. In contrast, divalent ions like calcium (Ca) and magnesium (Mg) show slower removal rates due to their larger ionic sizes and higher charges, making them less permeable in the ED process. The EC removal efficiency increases progressively, reaching 91.9% by the end of the process. As EC removal improves, the selectivity ratios of Na relative to K, Ca, and Mg increase, indicating that sodium is removed more efficiently as the treatment continues. This suggests that the ED system becomes more selective for sodium over time. The high permeability of sodium ions, followed by potassium, highlights the differences in ion mobility and selectivity in the ED process.

The effect on water quality was further assessed with varying ED operating parameters. Figure 15 depicts the impact of variable flow velocity (1 and 2 cm/s) and water recovery (50 and 80%). The provided graphs illustrate the flux (mg/m²·h) and removal percentages of various ions, including Na, K, Ca, Mg, Cl, NO_3, SO_4, and PO_4, under different conditions: baseline, water recovery (WR) 80%, and flow velocity (FV) 2 cm/s. For monovalent ions like Na and K, the flux remains relatively high across all

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conditions, with removal percentages exceeding 80%, indicating efficient ED performance. Na shows the highest flux and removal rate, especially under WR 80% conditions, suggesting enhanced efficiency at higher water recovery rates. Ca and Mg, being divalent ions, exhibit lower flux and removal percentages, reflecting their reduced mobility and permeability in the ED process. The second graph shows that Cl and NO_3 have the highest flux and removal rates, with Cl exceeding 90% removal under WR 80%, highlighting the ED system's effectiveness for these ions. SO_4 shows moderate flux and removal, while PO_4 has the lowest flux and removal percentages.

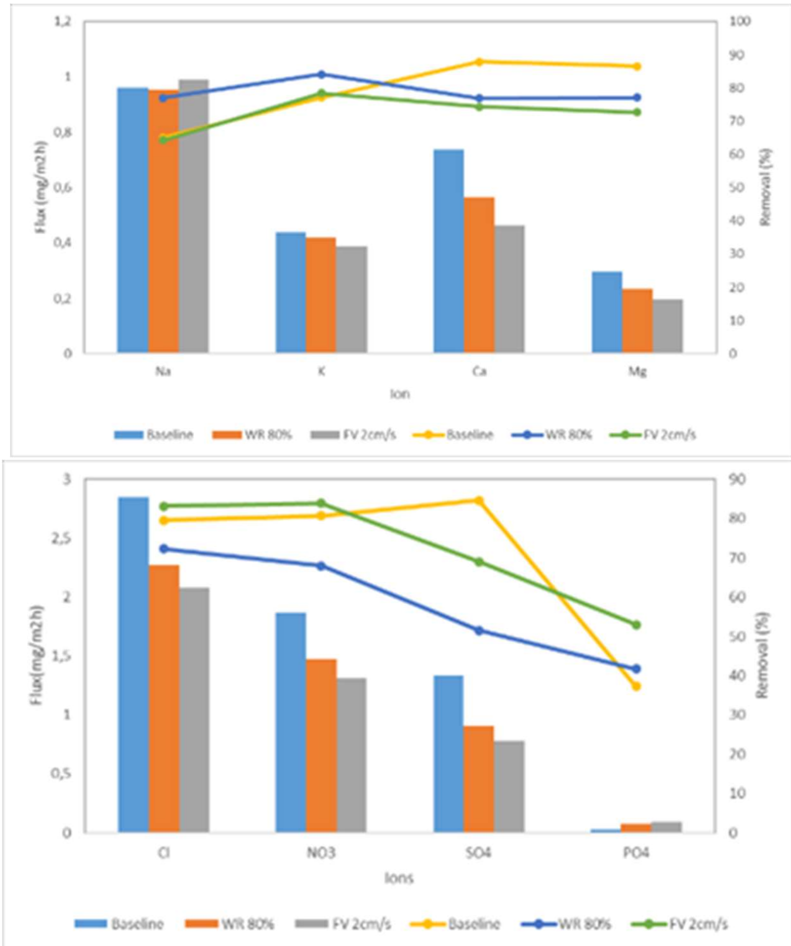

Figure 15. CS2: Effect of variable ED parameters - flow velocity (1 and 2 cm/s) and water recovery (50 and 80%) on ion flux and removal of the reclaimed water.

Figure 16 gives the specific energy consumption (SEC) for the reclaimed water at 60% and 90% water recovery. The SEC ranges between 0.12 -0.14 KWh/m^3. The nutrient concentration factors for recover are elaborated on in deliverable D1.5.

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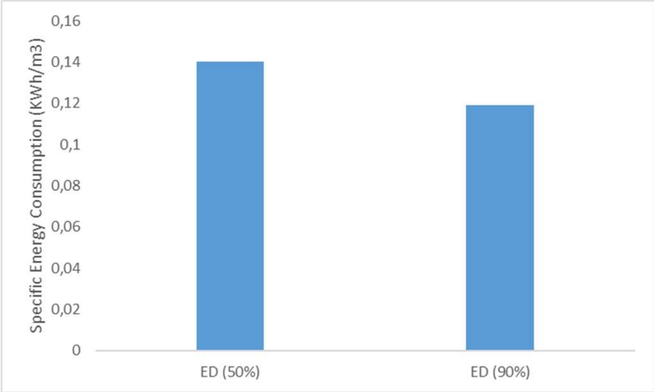

Figure 16. CS2: Specific energy consumption (kWh/m³) for 60% and 90% water recovery.

Capacitive Electrodialysis (C)ED Pilot

*A*n overview of the experimental conditions to determine the performance of the ED set-up is given in Annex 1. Figure 17 shows the conductivity removal and ionic water quality of the irrigation water for reuse (diluate) with the baseline conditions for reaching the target conductivities (EC in the range of 1-<0.2 mS/cm).

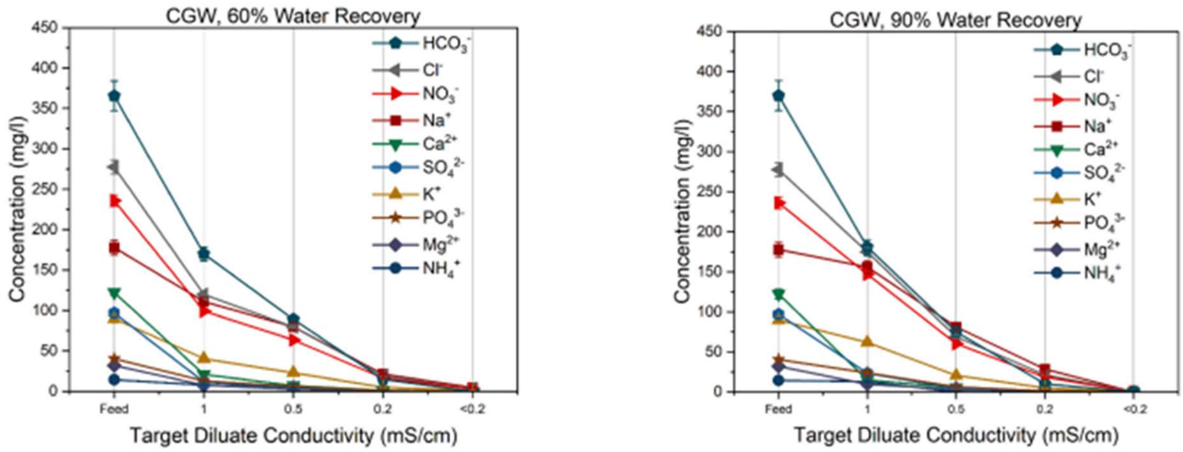

Figure 17. CS2: (C)ED ion transport and removal trends (60% and 90% water recovery, respectively).

Here, voltages beyond 12 V and 80 % water recovery showed diminishing returns in ion removal efficiency, indicating energy inefficiency due to back diffusion or electrode polarization. The most effective ion removal was observed at a crossflow velocity of 5.12 cm/s and with a 2-stage stack configuration. Furthermore, higher feed concentrations (2.5 mS/cm conductivity), from the range of greenhouse wastewater tested, showed improved ion removal. The study successfully achieved the highest target irrigation water quality across all feeds (conductivity 1- < 0.2 mS/cm, Na+ and Cl− < 0.1 mmol/L) with ions exhibiting variable removal efficiencies. Specifically, Na+ removal was less efficient compared to Ca2+ and Mg2+ (86 % ± 4 %, 97 % ± 2 %, and 98 % ± 3 % for diluate quality <0.2 mS/cm, respectively), which was significantly affected by increasing voltage and water recovery. In conclusion, (C)ED emerges as a promising alternative to traditional desalination techniques like RO, especially for

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producing fit-for-use irrigation water and recovering nutrients. Its advantages in terms of energy efficiency and tunable ion removal position (C)ED as a sustainable solution for water and resource recovery in greenhouse horticulture.

Key performance indicators

The demonstration and results show that there is potential in (C)ED as a sustainable solution for water reclamation from greenhouse wastewater. The results of this case study are summarised using the ULTIMATE KPI's in Table 21.

Table 21. CS2: Overview of the ULTIMATE KPI's for water reclamation for case study 2.

KPIs specified in GA	Achievable targets - Full scale
Reduction in freshwater use by wastewater reuse (20%)	Full scale implementation of the demonstrated technology, with the efficiencies observed in ULTIMATE, will result in: • **16% reduction of the primary freshwater input to the greenhouse consortium for irrigation purposes** 60 - 90% recovery of water from the greenhouse wastewater

Comparison of technologies/processes

For this case study, we do not apply multiple technologies. In the current situation, the effluent after treatment at site is discharged and not reused, resulting in a 100% loss. In contrast, implementing full-scale recovery from the effluent discharge based in the proposed scheme could achieve a recovery rate of 50-90%. However, this solution requires investment in additional treatment processes to facilitate reuse (additional details on the specifics are given in deliverable D2.2 – total cost of ownership (TCO)).

Comparison of baseline situation with ULTIMATE solutions

In comparison to the baseline situation with no water reuse (Figure 12), the ULTIMATE solution can achieve up to 16% reduction in freshwater consumption (compared to primary input to the greenhouses) and 60-90% water recovery (compared to recoverable greenhouse wastewater). Furthermore, both ED and (C)ED were successful in achieving good quality irrigation water (conductivity<0.2 mS/cm, Na^+<0.1 mmol/L) for reuse from De Vlot wastewater. Additionally, comparison with current widely used technology reverse osmosis (RO) was assessed for the (C)ED set-up (Table 19). The total specific energy consumption (SEC) of the (C)ED system ranged between 0.14 and 0.24 kWh/m^3 across different feed compositions. With optimal (C)ED operating conditions, this was 4-fold lower than the modelled RO system, and significantly lower than reported ED and RO studies. While RO systems achieve lower conductivity in the diluate and higher concentrate concentrations, (C)ED offers a

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superior energy-efficient alternative. The operational expenditure (OPEX) for ED, particularly the cost of maintaining ion-exchange membranes, constitutes about 10% of the total cost, with the majority of expenses attributed to energy consumption (>80%). This makes the process highly sensitive to energy prices. However, discussion with the greenhouse stakeholders revealed that the energy price for the sector may be 3 times lower than average energy prices for other sectors, making the application of this technology more attractive.

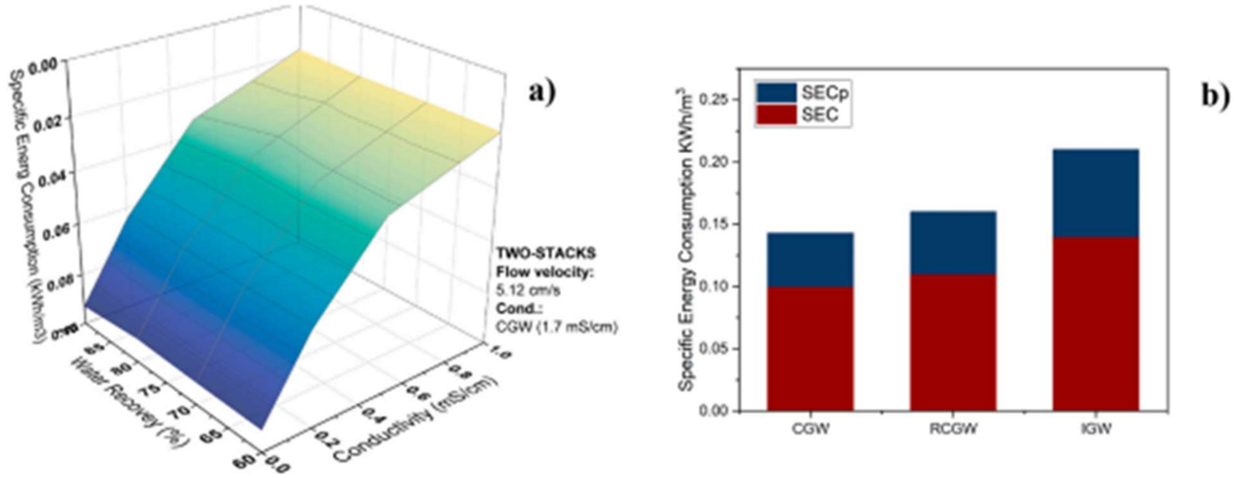

Figure 18. CS2: a) Specific energy consumption (SEC) in KWh/m³ for desalinating greenhouse wastewater (CGW feed composition (EC 1.7 mS/cm)) using (C)ED. The colour gradient from light green to dark blue represents an increase in SEC corresponding to target conductivities (1 to <0.2 mS/cm), achieved over water recoveries of 60–90 %. b) Total energy consumption (SEC stack and SEC Pump) for all compositions and 90 % water recovery.

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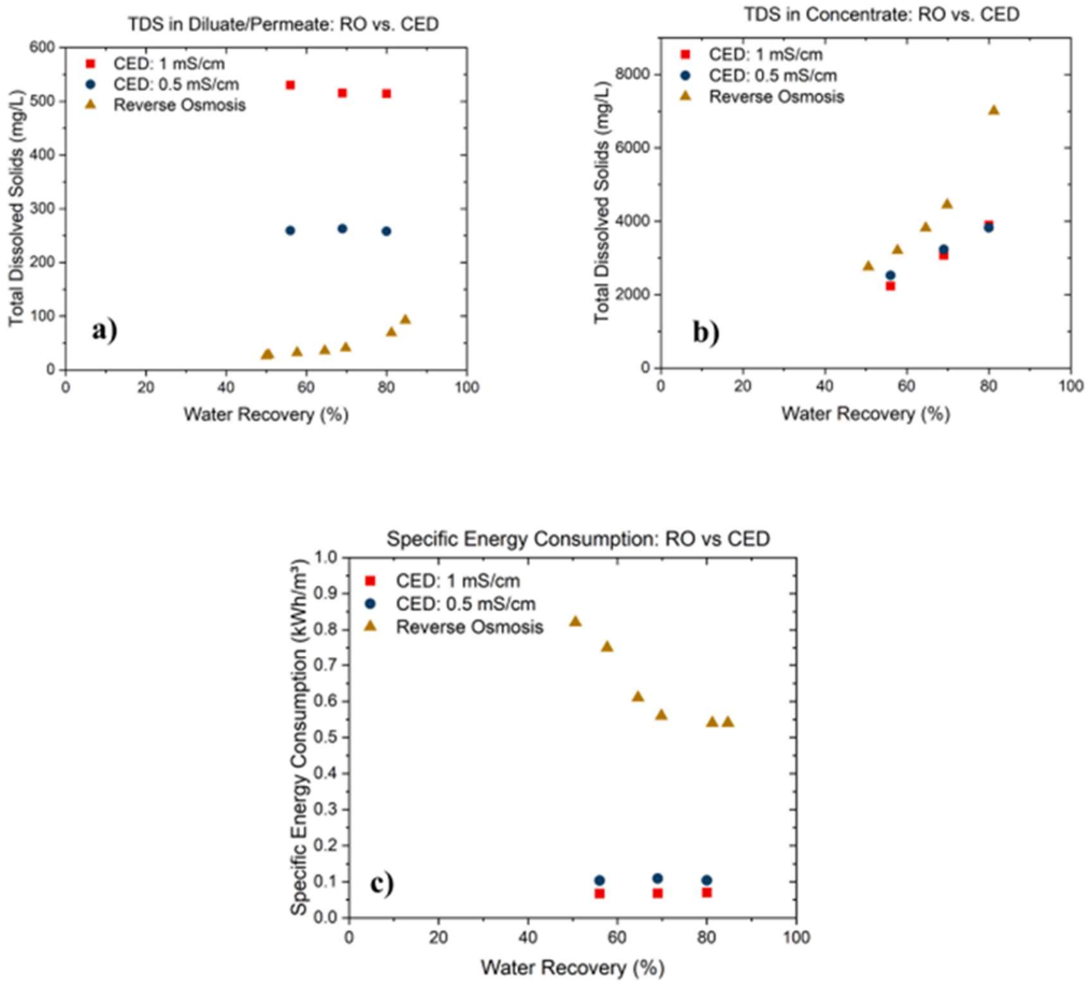

Figure 19. CS2: Comparative evaluation of capacitive electrodialysis (C)ED and reverse osmosis (RO) Technologies for a) ion removal performance, b) concentrate generated (TDS), and c) estimated SEC. Feed composition (EC 1.7 mS/cm) and crossflow velocity of 5.12 cm/s considered as operating parameters for both technologies.

Quantitative microbial Risk Assessment (QMRA) for plant pathogens

A risk assessment in relation to the presence and removal of plant pathogens for the reclaimed water was performed. This assessment was performed under the boundary condition that the recovered water will need to be treated by an additional disinfection step before application as irrigation water will be possible. This disinfection step was not demonstrated in ULTIMATE. As within the case study, the retention of plant pathogens and their transfer via reclaimed water is not estimated, quantitative microbial risk assessment (QMRA) approach is used. QMRAs are widely used for risk assessment from human pathogens, however for plant pathogens a standardized approach is missing. Figure 20 gives the proposed QMRA framework developed for plant pathogens, developed as part of this work.

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	Greenhouse wastewater	ULTIMATE Treatment	Treated output	Storage/distribution	Exposure Ultimate	Dose response	Risk characterisation
Data	Cpp Select 'index pathogens' Process indicator organisms Analysis method used	Which Tech? ED + Disinfection LRV of Tech for plant pathogens Process conditions	Cpathogens treated water	Pathogen fate Pathogen growth External contaminants Crossover spreading	Ways of irrigation (data available) Exclude pathogens soil Water considered Select	Define selected pathogens Which end point? Disease % of plants Loss of crops Economic loss Impact land	Crops loss by growers experience

Possible indicator pathogens	Group
Bacteria count	Bacteria
Mold plate count	Fungi
Yeast plate count	Fungi
Gray mold (Botrytis cinerea)	Fungi
Fusarium spp	Mold
Phytophthora spp	Oomycete
Pythium spp	Oomycete
Cucumber Green Mottle Mosaic Virus (CGMMV)	Virus
Tomato Brown Rugose Fruit Virus (ToBRFV)	Virus

Pathogen	Type of microorganism
A. rhizogenes	Bacteria
A. tumefaciens	Bacteria
Erwinia spp .	Bacteria
Cucumber green mottle mosaic virus (CGMMV)	Virus
Fusarium spp .	mold
Pythium spp .	oomycetes
Pepper mild mottle virus (PMMoV , March to June)	Virus

Figure 20. CS2: QMRA framework developed for plant pathogens and potential indicator pathogens from literature (above) and from samples analysed by analysis company (below).

One of the major challenges in developing a risk assessment for plant pathogens is the sparse availability of data on indicator plant pathogens. This includes information on their inactivation by various disinfection methods, transport pathways, and the quantification of potential outbreaks. Based on literature and expert experience, potential indicator pathogens were identified and are depicted in Figure 20. Additionally, most of the existing data pertains to plant pathogens from municipal wastewater, which is significantly different from greenhouse wastewater, further limiting its applicability. In conclusion, a QMRA framework is proposed for assessing

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the risk of plant pathogens in water reclaimed from greenhouse wastewater. This framework highlights the progress made so far and identifies critical data gaps that need to be addressed to achieve a comprehensive QMRA assessment for plant pathogens.

Fate of nutrients

In terms of nutrient recovery, the (C)ED technology used in the project can retain up to 30% of nutrients in the irrigation water for reuse. While it is effective in concentrating nutrients, further treatment steps are required for complete valorization. This is further elaborated on in deliverable D1.5.

2.2.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

The use of electrodialysis (ED) for water reclamation in greenhouse horticulture has provided several valuable lessons. Target irrigation water reuse conductivities of 1 to <0.2 mS/cm with 60–90% water recovery was successfully achieved using a lab-scale ED setup and a capacitive electrodialysis (C)ED pilot. This demonstrates that ED can meet stringent water quality requirements, essential for safe and effective irrigation. The deployment of selective membranes has proven effective, though the initial costs are high. However, these costs are often overestimated, and as technology advances, the costs and energy requirements are expected to decrease significantly. Future developments should focus on reducing these costs and improving energy efficiency to make ED even more viable. Despite the current higher costs and energy needs, the potential for high water quality and significant water recovery makes ED a promising technology for sustainable greenhouse operations.

Best practices and recommendations for technology design and operation in the symbiotic frame

Optimizing water reclamation in greenhouse horticulture using electrodialysis (ED) requires a comprehensive approach integrating best practices and supportive policies. Financial incentives, such as subsidies and low-interest loans for large-scale demonstrations, are crucial for encouraging the adoption of ED technology. Developing clear and comprehensive guidelines for water reuse, recognizing recovered nutrients as non-waste resources, and simplifying associated certifications will facilitate the integration of ED systems into greenhouse operations. Stimulating collaboration between academia and companies is essential for developing cost-effective membranes and other components. Third-party validation of circular economy technologies will build trust and credibility among stakeholders. Additionally, a stable and long-term legal environment is necessary to encourage stakeholder investment and protect against regulatory changes. By addressing these financial, regulatory, and

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collaborative aspects, the adoption of ED technology can lead to sustainable and efficient water and nutrient management in greenhouse horticulture.

Crucial factors for technology implementation and its optimal performance

- Successful implementation for water reclamation from greenhouse wastewater showcased with the case study. More full-scale demonstrations (Na^+ removal and water recovery) in collaboration with stakeholders are essential for market uptake
- Current costs of ion-exchange membranes and energy can be a bottleneck for uptake as compared to other technologies
- Discharge management (concentrate) is still a (technical & regulatory) issue
- Complete solution (incl. disinfection) still to be assessed (dealing with food products)
- Discrepancy in water demand and supply – buffering or storage (e.g. subsurface) required, legislation for such storage unclear
- Risk-averse nature of farmers/end-users, trust and incentives needed

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2.3. Monitoring, modelling, and control system to avoid high chloride concentrations in reuse water in Rosignano (CS3, IT)

2.3.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

The ARETUSA Consortium has been established in 2001 with the aim to associate an urban water utility (ASA Azienda Servizi Ambientali Spa), an industry (Solvay Chimica Italia Spa) and a technology provider (TME Termomeccanica Ecologia Spa) in a public and private partnership (PPP) to optimize water management at regional level.

Thanks to ARETUSA water reclamation facility, Solvay replaces high-quality groundwater with fit-for-purpose treated municipal wastewater for industrial use, while groundwater is more exploited for drinking water production to serve the coastal areas of Cecina and Rosignano.

Up to 3.8 Mio. m^3 per year of treated municipal wastewater is already reused by the industrial partner Solvay, freeing up private industrial wells for drinking water use.

Currently, the Solvay plant has highly expanded both in terms of production and variety, which further increases the water demand. The plant produces sodium carbonate, sodium bicarbonate (also for pharmaceutical use), calcium chloride, chlorine, hydrochloric acid, chloromethane, plastic materials, peracetic acid and hydrogen peroxide.

The ARETUSA water reclamation facility (Figure 21) has been operational since 2006, was designed to treat the secondary effluent coming from the two municipal Wastewater Treatment Plants (WWTPs) of Cecina and Rosignano by chemical, physical, and biological processes in order to reach the quality requirements of Solvay for industrial reuse. In this way the industry uses treated wastewater instead of withdrawn water from wells, making it available to ASA for drinking water production, generating added value for society and the environment.

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Figure 21. CS3: ARETUSA Water Reclamation Plant - Rosignano (Italy).

The Cecina WWTP is characterized by a final accumulation tank connected to the ARETUSA plant via an underground pipeline of approximately 11 km. The Rosignano WWTP, however, being located near the ARETUSA plant, is characterized by a pipeline that works by gravity. On this pipeline there is a manual valve which allows the partial conveyance of the effluent from the Rosignano plant to the equalization tank at the head of the ARETUSA plant. The ARETUSA plant was sized considering 50% of wastewater coming from the Cecina WWTP and 50% from Rosignano. This is because the WWTPs are located in territories that are very similar to each other (coastal cities, a few km from the sea), therefore it was agreed that the waters were characterized by very similar quality.

The sewage systems of Cecina and Rosignano are therefore affected by relevant intrusion of the salt wedge, which determines an increase in the concentration of chlorides and in the conductivity entering the WWTPs of Cecina and Rosignano. As these plants are urban WWTPs, the treated wastewater must comply, with the Italian Legislative Decree 152/2006, which provide maximum limits of the main macropollutants (COD, BOD5, TSS), being unnecessary to remove chlorides to accomplish with the regulation, Hence, they have not adequate technological sections (such as filtration and reverse osmosis) to remove them. Consequently, the effluents of Cecina and Rosignano WWTPs do not variate their concentration of chlorides significantly, so that the treated waters entering the ARETUSA reclamation plant are characterized by an important concentration of chlorides which determines an increase in conductivity, with peaks of up to 5000 µS /cm.

Description of the technology, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

CS3 aims at extending and optimising the quality and quantity of the treated water increasing the technical, economic, and environmental sustainability of industrial reuse, in a local circular economy context. To reach this purpose, the research

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activities of ULTIMATE project aim at developing a real-time data driven monitoring and process control system for seawater intrusion and infiltration to overcome salinity peaks in the influent to ARETUSA plant with the goal to increase water reuse.

To optimize the quality of treated water for industrial reuse by reducing the concentration of chlorides, the ULTIMATE project aims to develop a predictive system of the quality and quantity of sewerage networks starting from its modeling, the development of smart equalization system which, based on the quality of the wastewater, prioritise the entrance to the ARETUSA plant from Cecina WWTP effluent rather than Rosignano and vice versa (the logic will be shown below). Finally, the project involves the development of a matchmaking platform, which consists in determining the reuse of treated wastewater based on quality, i.e., if the conductivity of the effluent from ARETUSA complies with Solvay's requirements, industrial reuse will be proceeded. Otherwise the water will be used for irrigation.

Sewage system model

The sewerage network of the municipality of Rosignano is of a separate type and contains 12 pumping stations that convey the wastewater to the municipal WWTP. The municipality of Cecina is characterized by a mixed sewerage network, the wastewater is conveyed to the WWTP that serves the municipality through 15 pumping stations. For both municipalities, the diagram of the sewerage network is shown (Figure 22) with the length of the main sections and the corresponding diameters and with an indication of the main sewerage infrastructures (pumping stations and spillways).

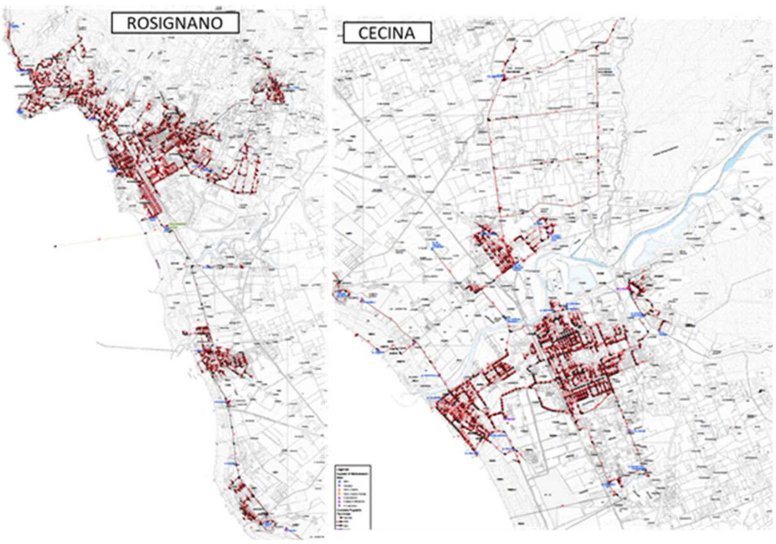

Figure 22. CS3: Diagrams of the sewerage networks of Cecina and Rosignano.

To understand where the intrusion was the greatest, the sewerage networks of Cecina and Rosignano were modelled using the EPA's open-source software, Storm Water Management Model (SWMM). These models were able to provide both the quantity and quality of the water entering the WWTPs.

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Early Warning System

The Early Warning System (EWS) involves the development of a system that stores the conductivity data, measured in real-time by a set of sensors installed along the network. In addition to this, based on this data, when the conductivity in the network exceeds the limit of 2000 µS/cm, the EWS will provide alert messages. The input data consists of conductivity measured at the plants and on the sewerage network, in particular in proximity of the Wastewater Treatment Plants (WWTP) of Cecina and Rosignano.

A statistical approach was adopted. This approach is capable of providing a probability of exceeding a pre-established conductivity threshold at the ARETUSA plant instead of a quantitative prediction.

Smart Equalization

The smart equalization system is based on the data that feeds the digital platform. Below it can be found the exemplary logics on the basis of which the smart equalization control algorithm was built.

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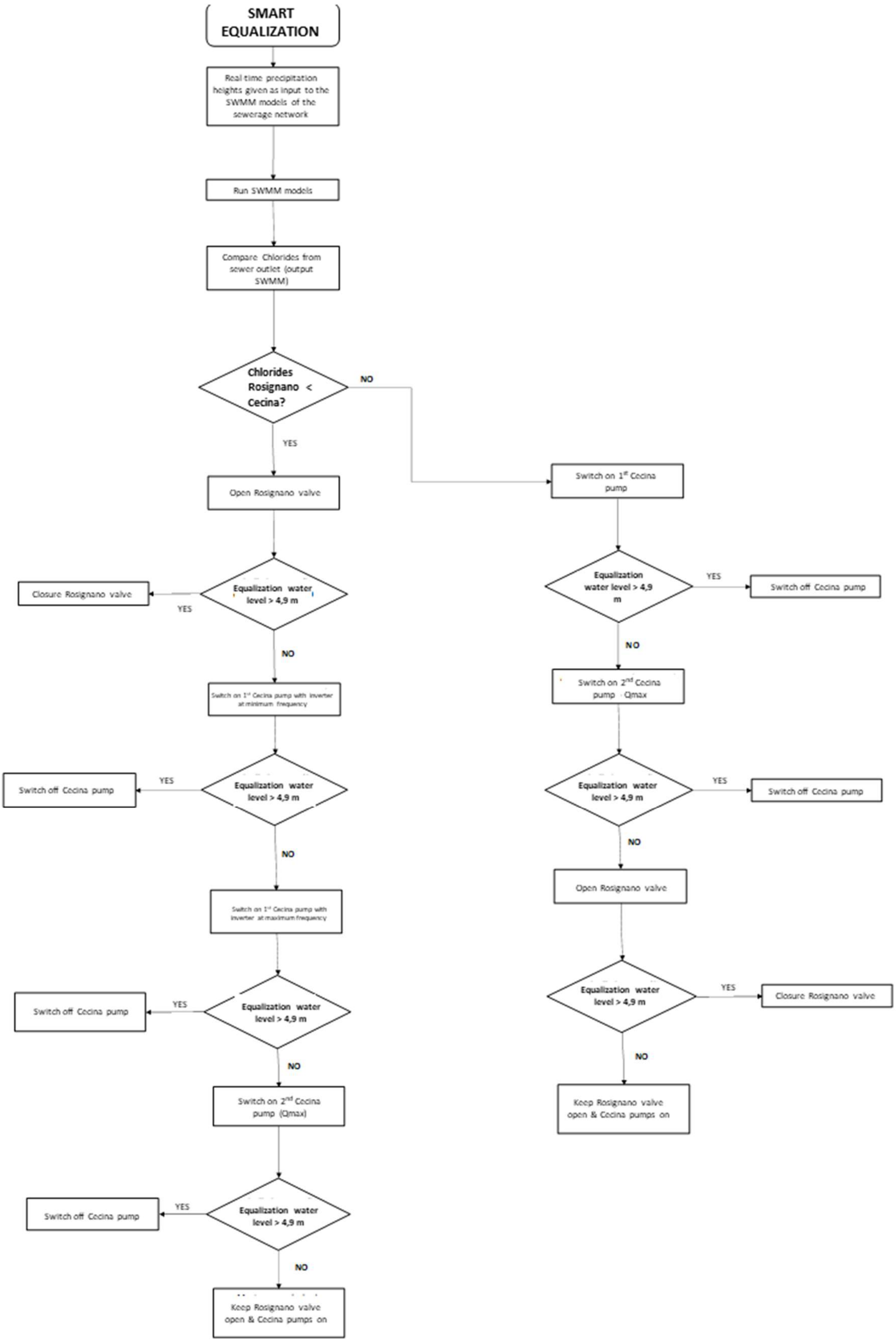

Figure 23. CS3: Smart equalization system logics.

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MatchMaking Platform

The Matchmaking platform is a decision support tool that aims to identify the most suitable application for reuse of the water treated in the ARETUSA reclamation facility. It is especially useful in case that industrial reuse is not possible due to high conductivity, or excess of reclaimed water is produced (reclaimed water production > industry requirements). In particular, the platform considers two macro categories: i) industrial reuse; and ii) agricultural reuse. Each category is in turn divided into subcategories with an associated threshold value of conductivity. For industrial reuse, the tool always tries to satisfy the water demand of the company, i.e., maximum of 433 m3/h. Only the flow in excess is used for agricultural purposes. When the water produced in ARETUSA does not satisfy the amount required by the industry and contains conductivity over 2000 µS/cm (which is the threshold to be used by the industry), the second option is to post-treat part of the effluent water by reverse osmosis (RO). In particular, if the input conductivity is between 2000 and 2900 µS/cm, 33% of the water coming out of the WRP will go to RO, whereas the rest is directly sent to Solvay industry. When the reclaimed waters conductivity is in the range 2900 - 5600 µS µS/cm, 66% of it is treated in reverse osmosis. Otherwise, all the water goes to osmosis (Figure 24).

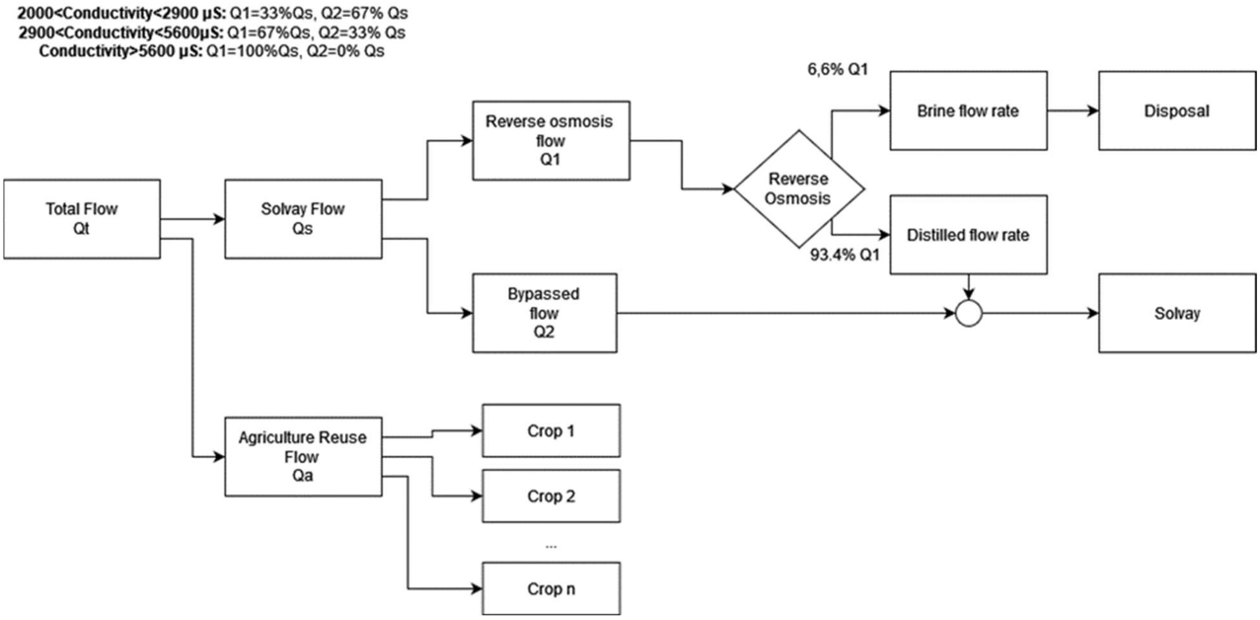

Figure 24. CS3: Logic of matchmaking platform.

Technical requirements for its implementation and operating conditions

The technical equipment necessary to implement the case study mainly consists of the sensors to measure wastewater flow and conductivity in the sewerage networks of each municipality and the reverse osmosis system. Other elements such as the smart equalization system and matchmaking platform do not require relevant technical issues. Their main novelty lies on their digital programming and language.

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To implement the hydraulic models, flow sensors were installed at the points: (i) Pista Ciclabile, Ramo Nord, Sileoni, Galoppatoio, Sileoni, WWTP (Cecina), (ii) Telesio, Polveroni, P3 (Rosignano), receiving data with high temporal resolution (5 minutes). In addition, conductivity sensors were installed in the following points: Porticciolo (Cecina), Caletta (Rosignano), Scoglietto (Rosignano), Mazzanta (Rosignano), Outlets of Cecina and Rosignano, and Inlet of ARETUSA Plant.

A reverse osmosis pilot plant was developed to improve the quality of the reclaimed water produced in ARETUSA. The pilot is fed by the effluent stream of ARETUSA, in particular from the pre-UV section. The pilot consists of a combination of 20-micron cartridge filter, an ultrafiltration section, followed by a $2m^3$ storage tank to backwash the UF, a 2-micron cartridge filter and two reverse osmosis membranes (Figure 25). In addition to understanding the ideal percentage to achieve the conductivity required for industrial reuse, the goal of this RO is to study the variation in energy consumption.

Figure 25. CS3: Reverse osmosis pilot plant.

2.3.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

Storm water management model (SWMM)

The SWMM models were validated in the dry period of the WWTPs, comparing the flow rates and concentrations provided by the model with those indicated by the data available at the plant input for the same simulation period.

Following this, the contribution of the salt intrusion was assessed. For this purpose, the SWMM model was integrated with the original model developed in Python. To do this, the input of the SWMM model was translated into Python language in order to modify/introduce specific parameters, including:

- Flow hydrographs: hydrographs have been inserted at specific nodes of the pipeline

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- Infiltration flows: these flows have been entered as specific values for the length of the pipeline
- Pump switching levels
- Precipitation events

The model was then operated thousands of times, modifying the parameters introduced above in order to identify the function that correctly described the infiltration flows in the network from both a quantitative and qualitative point of view (Table 22).

Table 22. CS3: Cumulative Qzb, Szb values for particular virtual point (for 24 hours) from optimization in Cecina.

Point	Qzb	Szb				L(Cl)	L (COD)	L (NH_4-N)	L (TSS)
		Cl	COD	NH_4-N	TSS				
	$m^3 \cdot d^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$
P1	9	1000	10.5	0.1	5	9	0.1	0.00	0.0
P2	10	1000	10.5	0.1	5	10	0.1	0.00	0.1
P3	29	1654	10.5	0.1	5	50	0.4	0.00	0.2
P4	32	1735	10.5	0.1	5	54	0.3	0.00	0.2
P5	35	1781	10.5	0.1	5	59	0.3	0.00	0.2
P6	90	2230	10.5	0.1	5	200	1.6	0.01	0.5
P7	169	2241	10.5	0.1	5	380	1.8	0.01	0.9
P8	97	2365	10.5	0.1	5	244	0.5	0.01	0.5
Total	471					1007	4.9	0.03	2.4

Table 23. CS3: Cumulative Qzb, Szb values for particular virtual point (for 24 hours) from optimization in Mu-1.

Point	Qzb	Szb				L(Cl)	L (COD)	L (NH_4-N)	L (TSS)
		Cl	COD	NH_4-N	TSS				
	$m^3 \cdot d^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$
P1	320	1420	10.5	0.1	5	454.0	3.4	0.03	1.6
P2	276	1000	10.5	0.1	5	276.5	2.9	0.03	1.4
P3	251	1000	10.5	0.1	5	250.6	2.6	0.03	1.3
P4	251	1420	10.5	0.1	5	355.8	2.6	0.03	1.3

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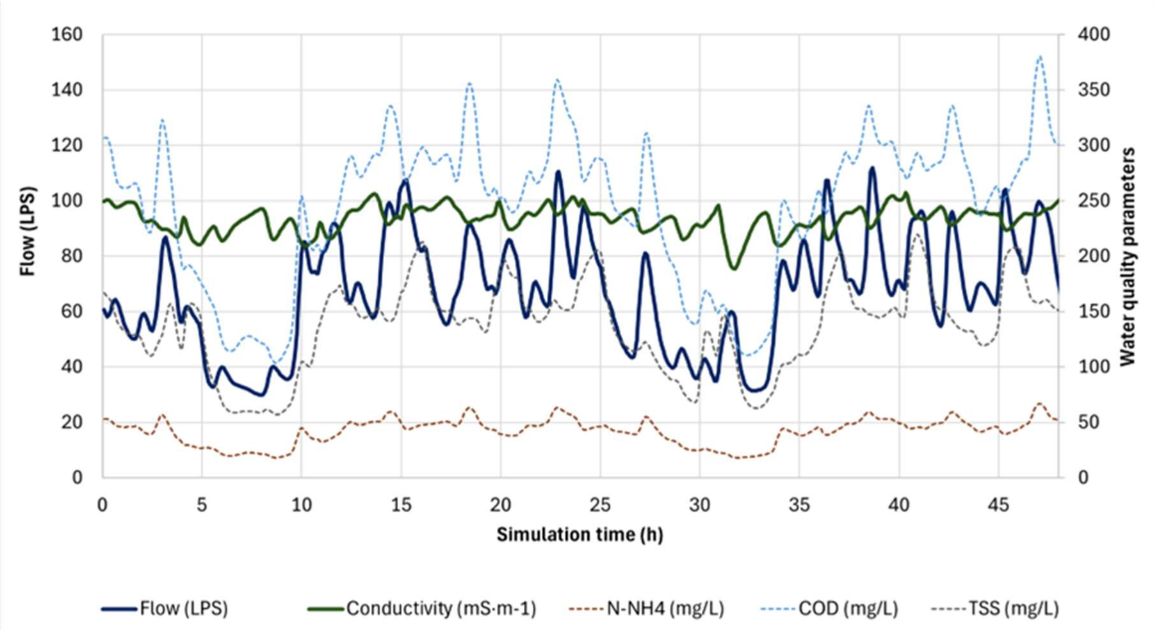
Point	Qzb	Szb				L(Cl)	L (COD)	L (NH_4-N)	L (TSS)
		Cl	COD	NH_4-N	TSS				
	$m^3 \cdot d^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$mg \cdot L^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$	$kg \cdot d^{-1}$
P5	130	1000	10.5	0.1	5	129.6	1.4	0.01	0.7
P6	302	1031	10.5	0.1	5	311.9	3.2	0.03	1.5
P7	328	1125	10.5	0.1	5	369.4	3.5	0.03	1.6
P8	276	1125	10.5	0.1	5	311.0	2.9	0.03	1.4
P9	276	1125	10.5	0.1	5	311.0	2.9	0.03	1.4
Total	2411					2770	25.3	0.24	12.0

Qzb: Infiltration flow in the simulated sub-catchments.

Szb: Wastewater characteristics in the simulated sub-catchments.

This way, it was possible to obtain a predictive tool capable of identifying the volumes and loads entering to the WWTPs and, subsequently, to the smart equalization system, considering the negligible reduction of conductivity and chloride concentration at the WWTPs. It is thus possible to start from such data to give input to the smart equalization system. For 2-d simulation (spring time), the results obtained are shown in Figure 26.



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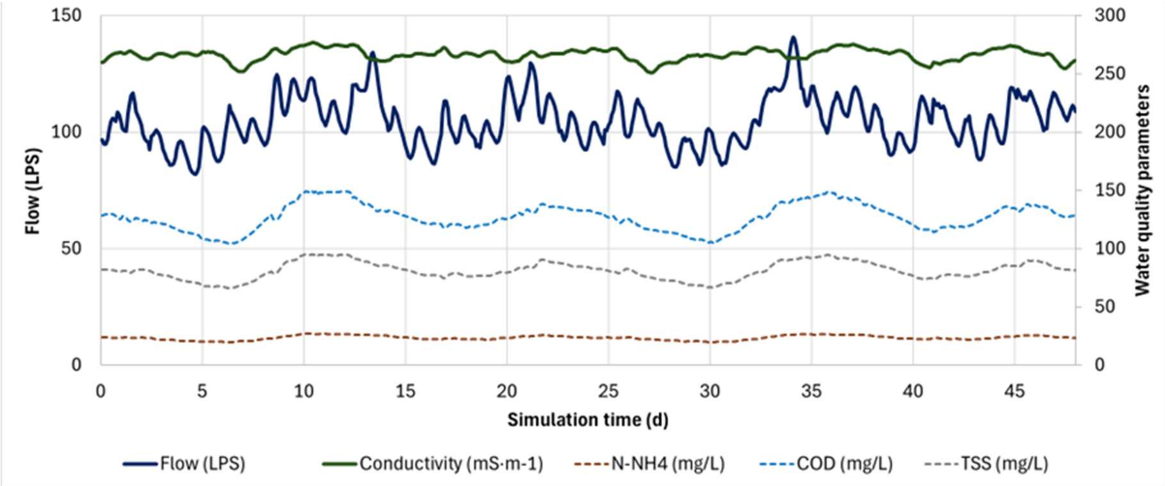

Figure 26. CS3: Quantity and quality of the sewage entering: a) Cecina; b) Rosignano.

Smart equalization

From the results of the simulation, it was observed that the conductivity of the mixed flow from Cecina and Rosignano WWTPs was over 2500 μS·cm^{-1} during most of the simulated period (Figure 27). But the amount of water discharged only accounted for an average of 19%. This was mainly due to the fact that the equalization system tends to prioritize the reuse to satisfy the water requirements of the industry. On the other hand, higher peaks of conductivity (>2600 μS·cm^{-1}) generally coincided with the higher peaks of discharge.

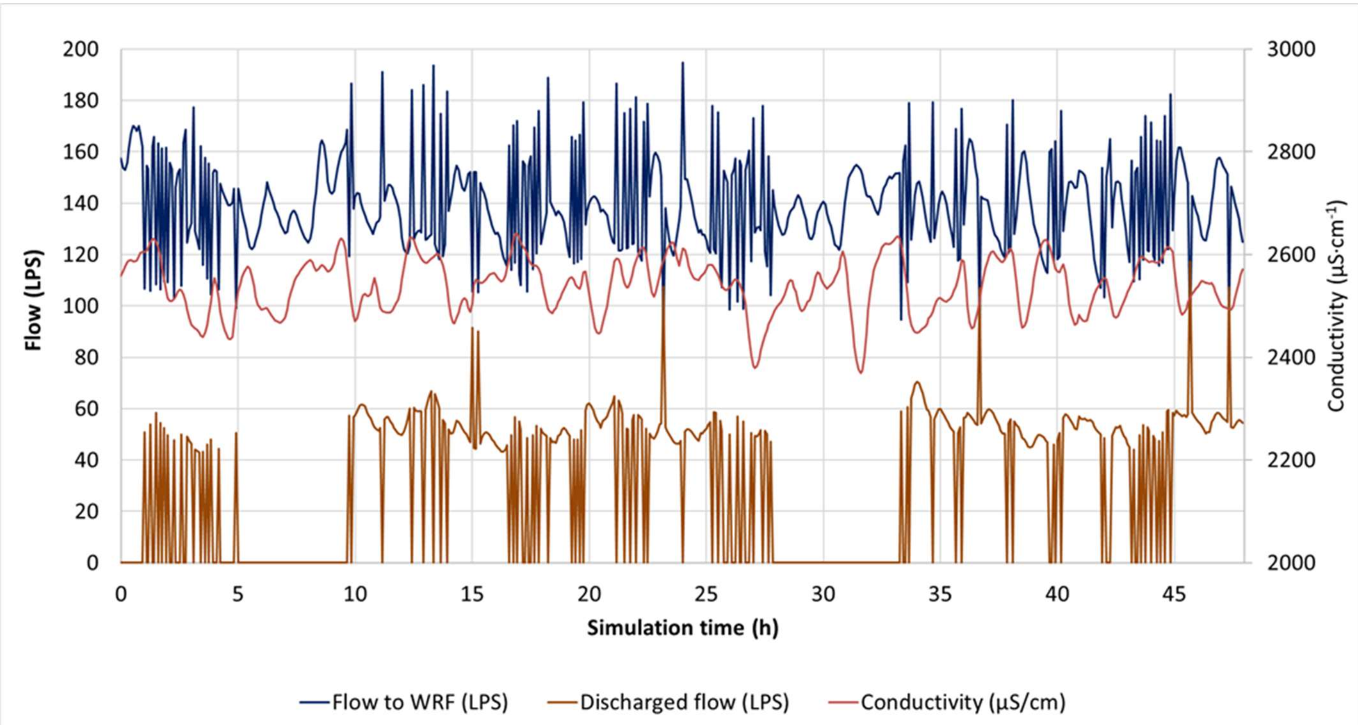

Figure 27. CS3: Distribution of the water flows carried out by the smart equalization system.

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Reverse Osmosis

As the smart equalization system alone is not able to allow the achievement of quality objectives required for industrial reuse, it is necessary to implement a reverse osmosis unit to improve the quality of the treated water (reclaimed water).

The plant has been operated for 2 weeks, treating water with a flowrate in the range of 450-800 L/h (Figure 28). It is highlighting that the amount of brine was approximately the same than the amount of permeate. The plant was operated this way to avoid high consumption in the system, but this implies that huge amounts of brine containing high conductivity (Figure 28) must be managed.

Regarding the quality of the permeate, this was considerably high, showing negligible conductivity during the whole period (Figure 29). Producing this high-quality permeate will enable to dilute the reclaimed water coming from ARETUSA containing high salinity (i.e., over 2000 μS/cm, conductivity in Figure 29), thus reaching the requirements for industrial reuse, as it is explained in the next section.

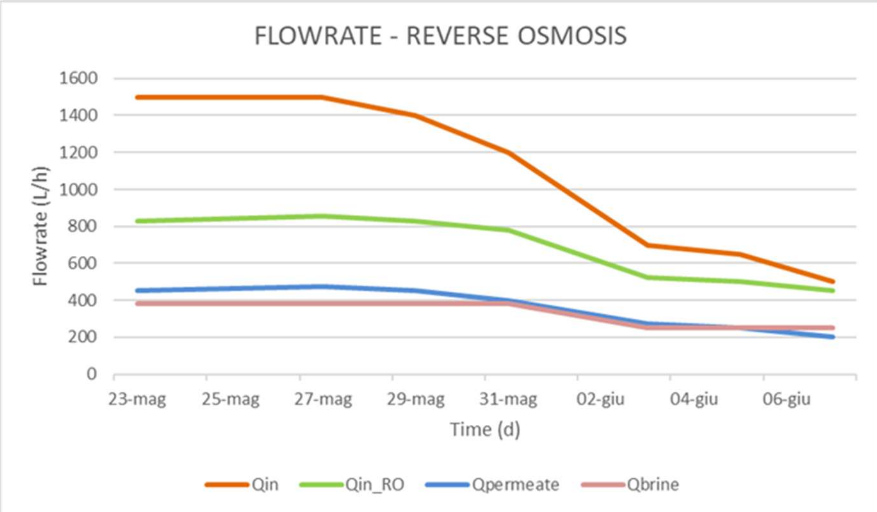

Figure 28. CS3: RO – Flowrates.

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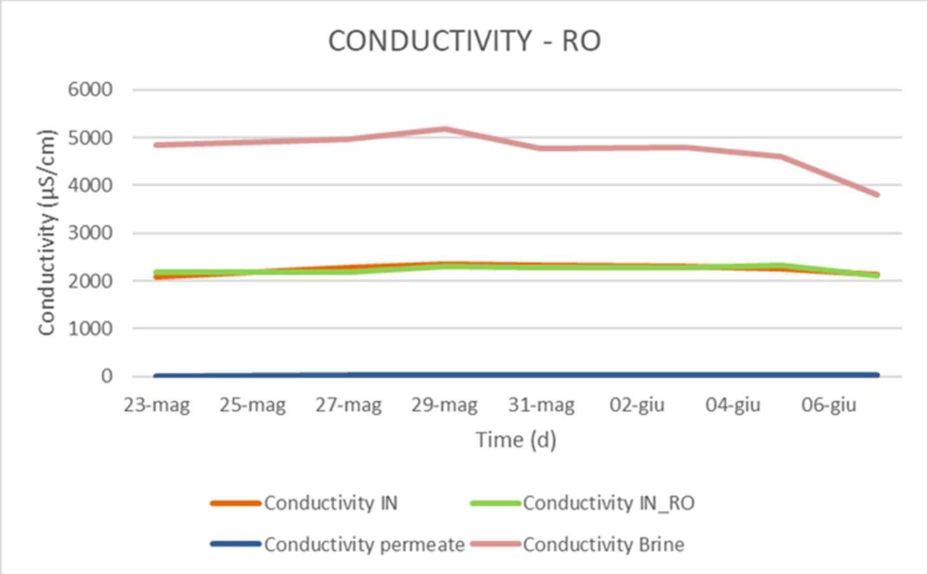

Figure 29. CS3: RO - Conductivity trend.

Matchmaking platform

The matchmaking platform aims to satisfy the water requirements of Solvay industry, so that the water that flows to both RO and directly to the industry was most of the time constant, their sum thus coinciding with the total requirement of Solvay (120 LPS). The extra flow was used in agriculture (Figure 30). But there were some moments where both flows to RO and direct industrial reuse showed several peaks of lower flows that coincided in time. During those short periods, the demand for industrial cannot be accomplished because the reclaimed water produced in the ARETUSA facility was not enough. Consequently, during those periods, all the water was sent to industrial reuse, avoiding agricultural reuse.

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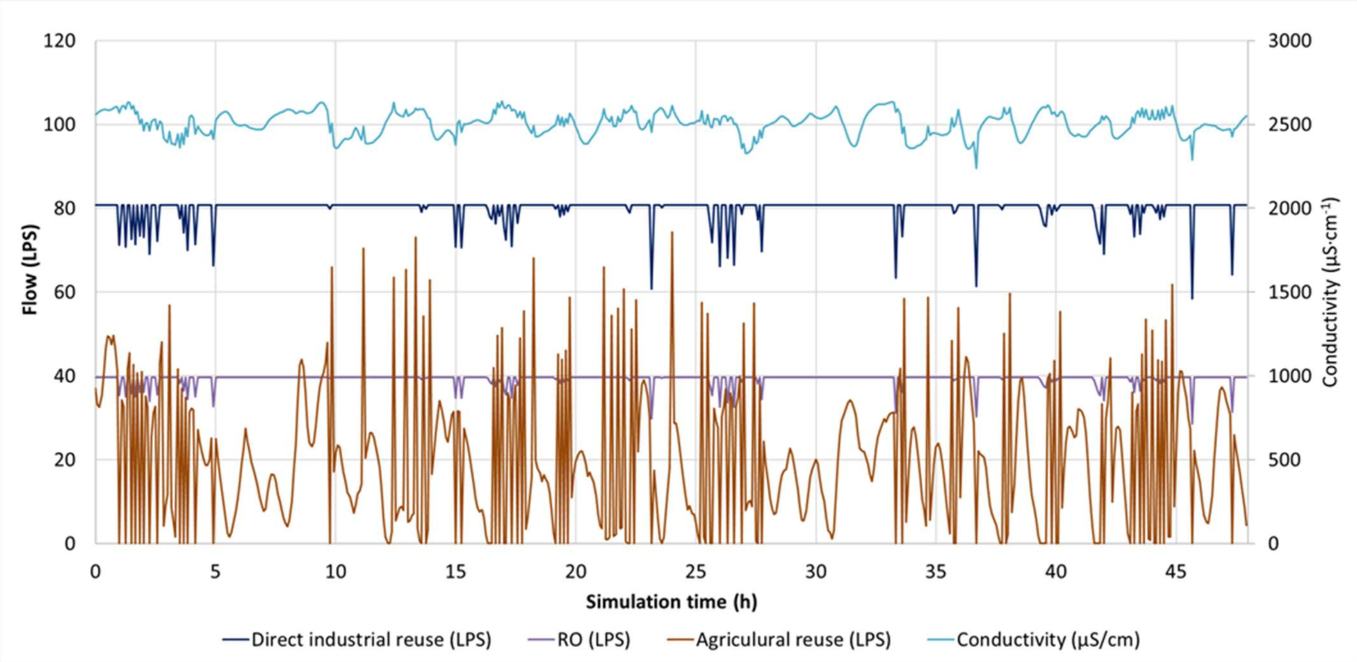

Figure 30. CS3: Evolution of the reclaimed water flows distributed by the matchmaking platform.

Comparison of baseline situation with ULTIMATE solution

Before the implementation of ULTIMATE project, there were no sensors or instruments for measuring flows in the sewerage network. For the municipality of Cecina, a previous model of the sewerage network (only of the coastal part was created in the context of the Interreg "ADAPT" project. ADAPT planned to install six flow meters as depicted in Figure 31, but only three (Porticciolo, Sileoni and Galoppatoio) work correctly. Corrective actions were therefore necessary to restore the functioning of the other instruments.

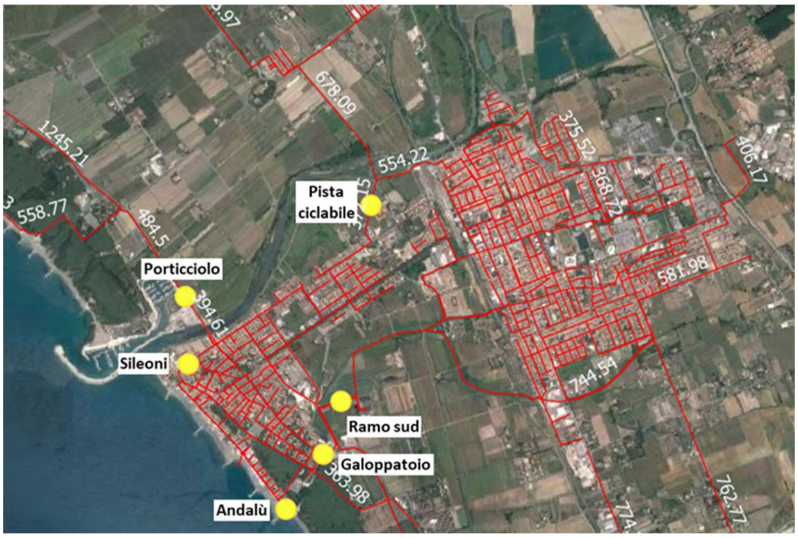

Figure 31. CS3: Flow meters already installed in the municipality of Cecina (of which 3 are reliable: Porticciolo, Sileoni and Galoppatoio).

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The work developed by ULTIMATE has allowed updating this system and install a set of flow meters and conductivity sensors. The data acquired by these sensors enabled to develop the SWMM models and the Early Warning system.

From the collected data, it was observed that due to the salt wedge, the background values of chlorides in the aquifer along the Rosignano and Cecina coasts have undergone a significant increase in recent years, as shown in Figure 32.

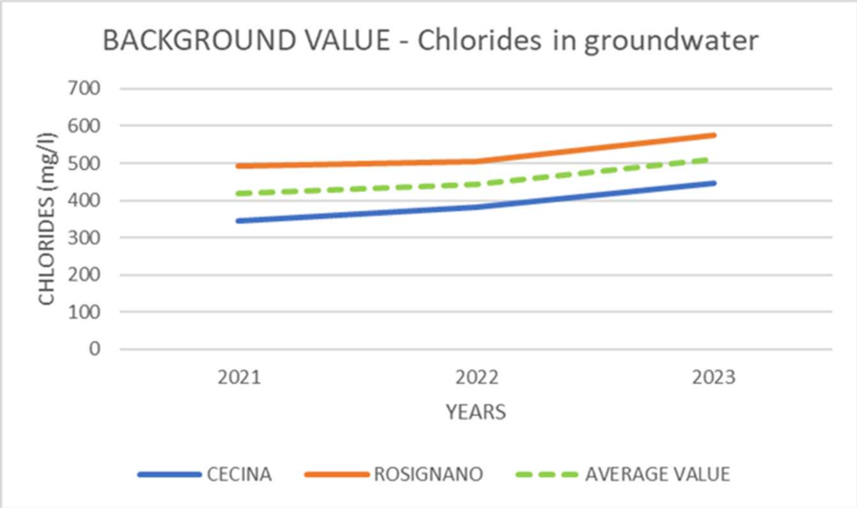

Figure 32. CS3: Background value - Chlorides in groundwater.

This situation poses serious issues to reuse the reclaimed water produced in ARETUSA, as the salinity content in this water is normally over the required limit (as explained before). To deal with this issue, a smart-equalization system was developed. This system deviates wastewater containing high peaks of salinity in order to avoid punctual salinity contamination. If this tool was not installed, two extreme situations would occur:

i) If only the 2000-$\mu S \cdot cm^{-1}$-conductivity threshold would be considered, all the wastewater produced during the whole simulated period would have been discarded, thus forcing the industry to satisfy their requirements for those days (i.e., 120 LPS) with groundwater.

ii) If no wastewater would be discarded, a total amount of 5568 m^3 of high-conductivity water would have been introduced to the system during these two days. This would have implied a 23% increase in the average conductivity of the water arriving to ARETUSA, which would entail higher post-treatment costs.

But, despite the installation of the automatic valve controlled by the equalization system, it was not possible to reach the optimal conductivity for industrial reuse. The trends for conductivity in the years 2021-2023 in Cecina, Rosignano and ARETUSA plants are shown in the figures below.

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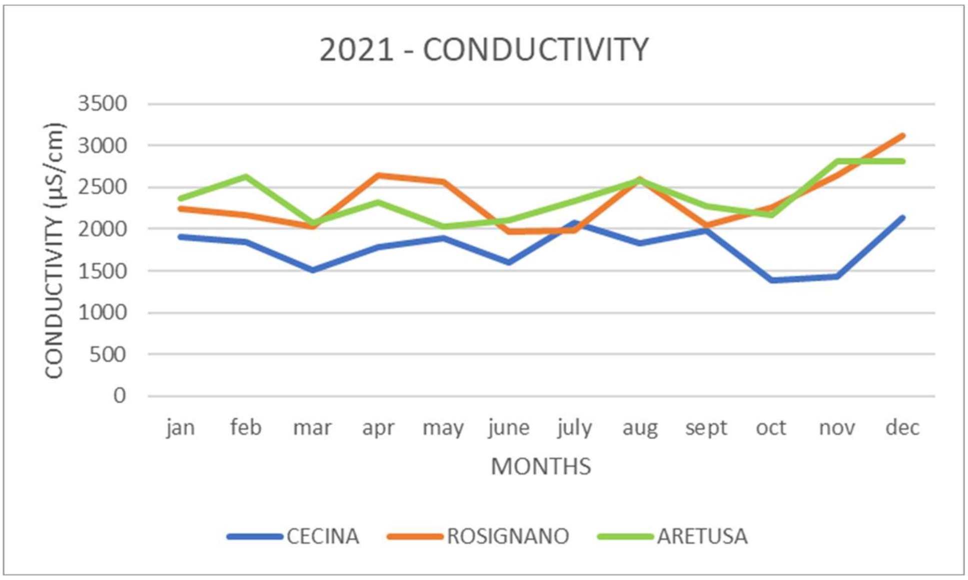

Figure 33. Conductivity trend – 2021.

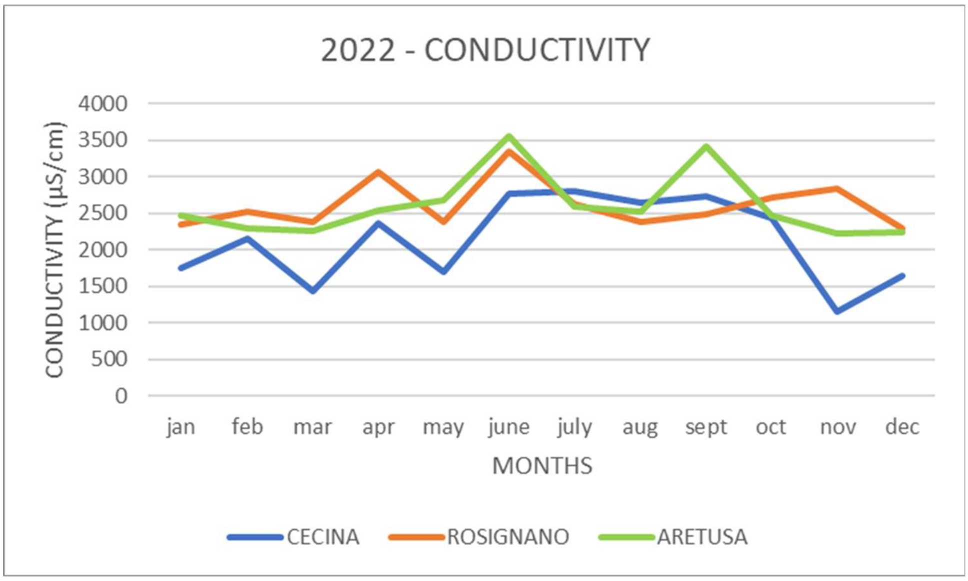

Figure 34. CS3: Conductivity trend – 2022.

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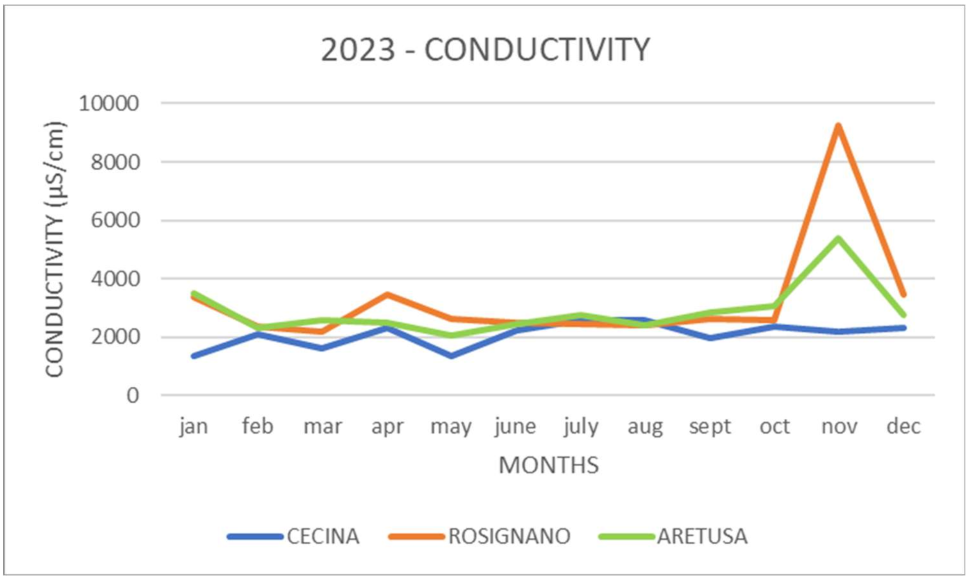

Figure 35. CS3: Conductivity trend – 2023.

Observing this trend, during the development of ULTIMATE project, it has been planned to evaluate the installation of a RO system, which was not planned at the proposal stage. Preliminary results have shown that conductivity is completely lowered in the permeate, as well as chlorides that decrease from 400-500 mg/L to <10 mg/L. However, initial cost calculations showed values of 0.33 €/m^3. This cost is considerably high for industrial reuse. Consequently, the water treated in this RO system is to be used in specific situations. In the case of this study, the reverse osmosis enabled to increase the water to be reused for industrial purposes as it can dilute the water that is directly sent to the industry. This implies that by only treating 40 LPS of reclaimed water in the RO process, it could be reused a total of 120 LPS for industrial purposes. If the RO would not be used, the reclaimed water would not achieve to the standards for industrial reuse so, it would be sent for agricultural reuse. This solution, despite being economically and environmentally efficient in certain cases, would not be the ideal alternative for this case study because if the company cannot receive reclaimed water from the WRF or the reverse osmosis process, they would need to consume high-quality groundwater, which is getting scarcer and scarcer in the area. It has to be also noted that the use in agriculture of this reclaimed water with high salinity must be restricted as excessive supply of salts will decrease the crop production due to the limited tolerance of certain crops to high levels of conductivity, also reducing the quality of the soil.

2.3.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

Industrial symbiosis approach was established between an industrial company and a water utility to prioritize the reuse of urban wastewater for industrial purposes. This

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requires low-salinity water, but the area of this case study is frequently affected by saline intrusion, thus creating water-related conflicts between the different economic activities. To reduce the adverse effects of saline intrusion in this area, a digital solution was proposed. It combines dynamic simulation model that predicts seawater infiltration and runoff with digital tools, i.e., smart equalization (control algorithm) and matchmaking platform (decision support system). The proposed solution aims to predict the periods where significant peaks of salinity enter to the sewage networks of the municipalities of Cecina and Rosignano. The tools aim to distribute the wastewater and reclaimed water streams to diverse applications (industrial, agricultural) and/or treatments (conventional treatment, reverse osmosis) to maximize the amount of wastewater reused in efficient and sustainable way.

The storm water management model (SWMM) models developed in this study were able to predict with relatively high resolution both the quantity and quality of the seawater infiltrated into the sewage networks of the municipalities evaluated. In both municipalities, seawater infiltration seemed to be a relevant factor affecting the sewage entering the urban WWTPs. In fact, the simulation carried out in this study showed wastewater conductivity remained in the range 2100-2700 μS/cm, which is over the established limit for industrial reuse. To avoid excessive conductivity entering the reclamation facility, the smart equalization system developed in this study enabled to discharge 19% of the total with the aim to reduce salinity loads by 23%. From the total reclaimed water produced, 87% was sent to the industry and the remaining 13% would be used for irrigation. With respect to agricultural reuse, its reusable capacity will depend on the type of crop present in the area and its ability to tolerate water with a certain conductivity. Depending on the final conductivity of the reclaimed water used in agriculture, the platform will indicate the percentage of tolerance of the selected crop(s).

Best practices and recommendations for technology design and operation in the symbiotic frame

ARETUSA PPP is a good example of industrial symbiosis practices, where urban wastewater management and the chemical industrial sector complement to each other to improve circularity in water.

The results obtained in ULTIMATE give evidence on the potential use of the combination of hydraulic modelling and digital tools to increase the reclaimed water production from wastewater treatment and reuse facilities affected by saline intrusion.

The approach implemented in this study can be very useful for replication in coastal areas where saline intrusion is relevant.

For developing accurate hydraulic models that aim to detect peaks of saline intrusion, it is of highly importance to acquire robust local data with high resolution. To this, the amount of installed sensors and their position must be adequate. The same issue can be extrapolated for the early warning system, the smart equalization system and

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matchmaking platform. Lack of data will significantly affect the performance of these digital tools.

Apart from the above, the critical situation of the region in terms of saline intrusion forces the reclamation plant to be updated with advanced treatment, i.e., reverse osmosis. Reverse osmosis enables to produce high-quality water than can be used to dilute the reclaimed water containing salinity that slightly overpass the requirements of the industry. But despite the benefits of RO in terms of water quality, their energy consumption is significantly high, so that it is essential to apply this kind of process only when it is necessary to minimize the economic and environmental impacts related to water reuse. Techno-economic and environmental assessment of the process is thus essential.

Next steps in this case study will include results from energy and environmental assessment to be coupled to the matchmaking platform (multi-criteria assessment). This way, the reclaimed water will be distributed to the different streams (industrial reuse, reverse osmosis, agricultural reuse or discharge) depending not only on the conductivity of the reclaimed water, but also on the economic and environmental impacts that each option will cause, with the goal to reduce them.

Future studies will also consider using reverse osmosis for agricultural reuse. In this case, the quality of reclaimed water does not need to be as high as in industrial reuse, so that the energy and environmental impacts would be much lower.

Crucial factors for technology implementation and its optimal performance

- Correct distribution of sensor for wastewater flow and conductivity is necessary to develop early warning systems based on modelling and digital tools.
- The saline intrusion is the region is getting more severe. Advanced treatment (reverse osmosis) of a fraction of reclaimed water is necessary to achieve the required quality.
- The implemented solution (modelling + digital tools) have the potential to increase reused water in the region for both industrial and agricultural use.

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2.4. Reuse of fruit processing wastewater in Nafplio (CS4, EL)

2.4.1.Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

The eastern Peloponnese is one of the most productive regions in Greece in terms of citrus fruit (it is to be highlighted that Greece is the third largest producer of citrus fruit in the EU). Alberta S.A. is a Greek fruit processing industry and specializes in the production of fruit juice concentrates, fruit purees and concentrates, clarified juice concentrates, NFC juices, as well as tailor made products and blends, since 1981. Not only do they produce fruit juices but also vegetable juices, such as carrots and red beets. The majority of fruit juices come from citrus fruits (oranges, lemons, grapefruits, and mandarins), pome fruits (apples, pears), stone fruits (peaches, apricots), pomegranates, chokeberries, grapes, carrots and red beets.

Particularly in the Argolida area, where Case Study 4 is situated, there is an increasing water demand for irrigation. Most irrigation water comes from wells, often illegal ones. This practice, along with the high-water consumption of the fruit processing industry, is exerting great pressure to the local aquifer. Over-irrigation has led to subsequent intrusion of the seawater into the aquifer, which has reduced the quality of the groundwater aquifer, which exhibits high conductivity values (in the region of 3000 μS/cm). With a view on reducing the overall cost of disposing wastewater to the municipal biological treatment, as well as meet the effluent legal criteria, all sizeable fruit processing plants of the area have constructed and are currently operating individual primary biological plants. Nonetheless, each plant periodically ceases to operate as a result of the seasonality of the production. This practice increases operational costs, as it is necessary to kick start the wastewater treatment plant when it is again needed.

Alberta S.A has a primary biological treatment plant of about 20 m^3/h capacity to meet the effluent criteria, as well as to reduce the cost of disposing wastewater to the municipal WWTP. This process is mainly focused on the removal of organic matter, achieving more than 90% removal (COD concentration in the outlet stream).

Before ULTIMATE, there was no communication let alone an established symbiotic system among the water stakeholders in the area, which could have enabled water reuse or recovery of any valuable resources.

In the frame of ULTIMATE, Greener than Green (GtG) Technologies developed VesperX, a mobile unit that treats and recycles water and nutrients present in aqueous industrial by-products.

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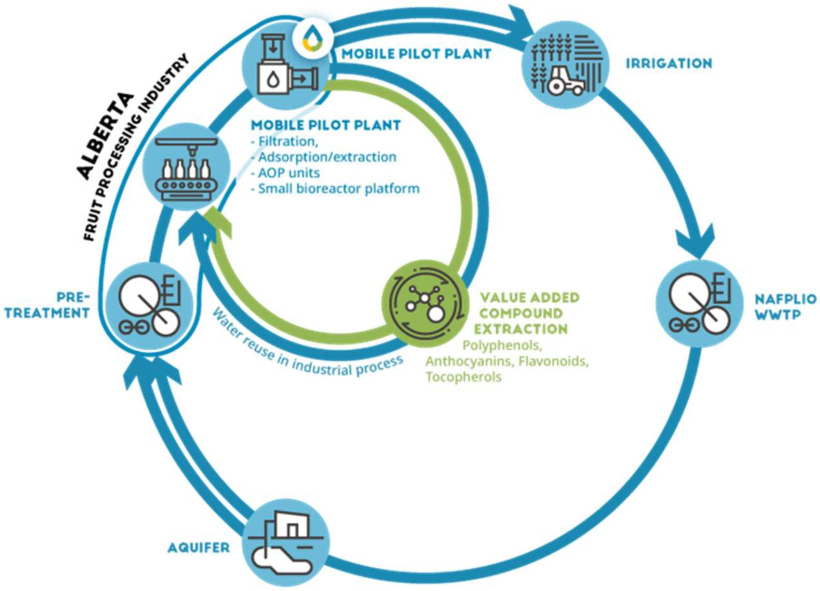

Figure 36. CS4: Schematic presentation of the case study.

The by-products of fruit and vegetable processing sector contain a significant number of valuable compounds. A certain class of such compounds, polyphenols, are complex organic molecules with significant biological activity and as a result high market price. Their complex structures make their synthesis indeed very challenging leaving nature as the only plausible source. Their price per gram in their purest form ranges from a few euros to hundreds of thousands of euros.

Figure 37. CS4: VesperX unit.

VesperX combines a number of physicochemical and biological processes to isolate, value-added compounds and treat the remaining water, so the extent that it is rendered suitable for irrigation and/or secondary industrial uses.

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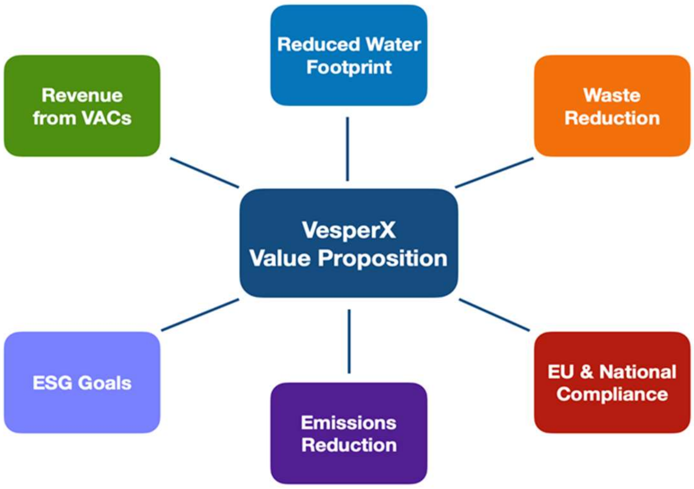

Figure 38. CS4: Value Proposition of VesperX.

VesperX is offered as a Product as a Service (PaaS) i.e. the units are not sold to the customer, they are leased for an agreed period. This model perfectly fits with the seasonality of the food processing sector and eliminates the need for extensive capital expenditure. It is needless to say that the revenue generated from the exploitation of valued-added compounds (VACs) significantly offsets the cost of the treatment unit, thereby self-funding the environmental operations.

VesperX provides an opportunity for symbiosis not only within the water and materials clusters but also extends the notion of symbiosis to the societal aspect as an industry no longer competes for water with the weaker stakeholder: the farmers.

Description of the technology, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

A fully functional VesperX unit is installed at Alberta's production facility. Consisted of two 20-foot containers suitably converted into treatment tanks, and an ISOBOX containing the electrical installation, control units and advanced oxidation processes (AOP) module. The current TRL is estimated to be 6-7 the unit is capable of treating the water by-product of fruits & vegetables such as oranges, carrots, tomatoes and olives.

The processes that are employed, were initially developed on a lab scale. The overall process is described by the following layout (Figure 39):

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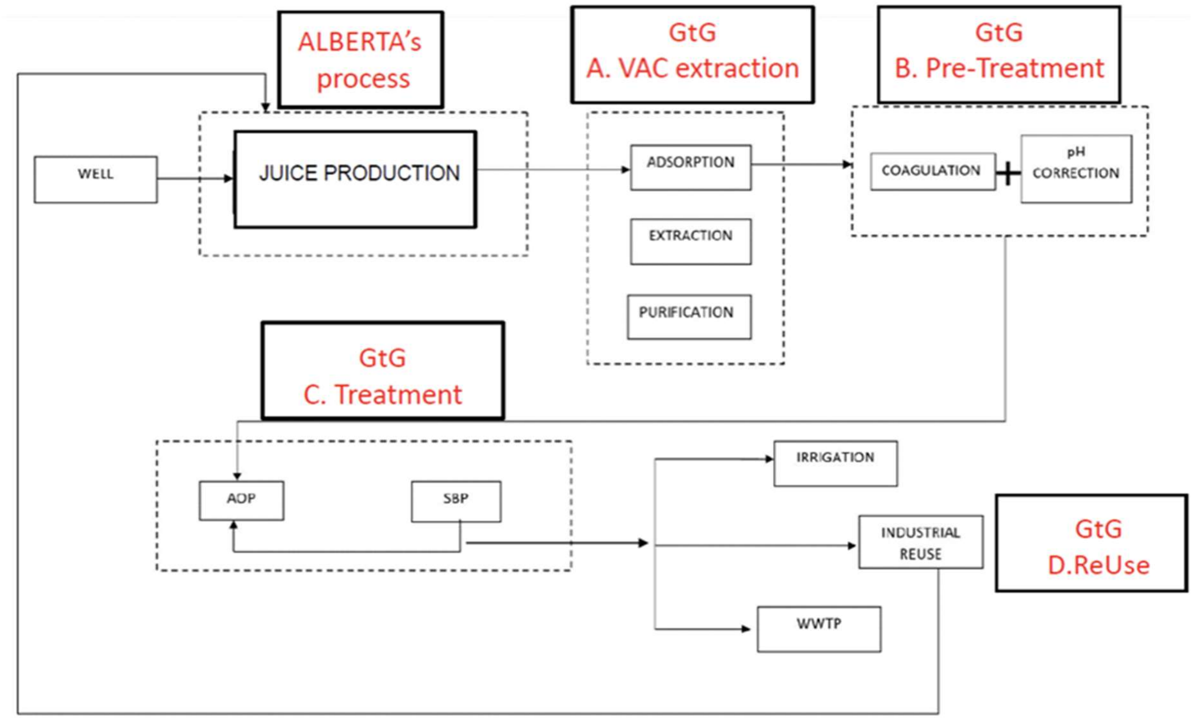

Figure 39. CS4: Layout of the pilot process deployed at Alberta S.A.

The pilot comprises of the following components:

A. VAC adsorption /extraction unit

In this module VACs are selectively adsorbed on a solid phase material that exhibits high affinity towards phenolic compounds relative to other chemical species. The extraction step deemed to take place prior to any other physical or chemical process. This module also addresses the objectives of subtask 1.4.3 (Recovery of high-added-value compounds in Nafplio). More details are presented there.

Based on the results of extensive laboratory experiments, the type of sorptive material, contact time and adsorption capacity, where selected and called up to fit the described pilot unit (Figure 40).

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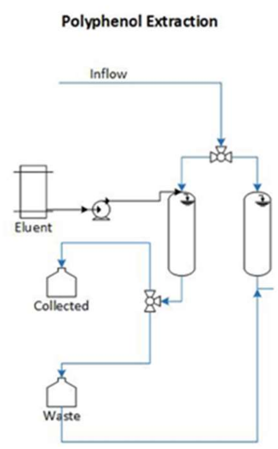

Figure 3. VAC adsorption process

Figure 4a. VAC adsorption lab-scale unit

Figure 4b. VAC adsorption pilot sub-unit

Figure 40. CS4: VAC adsorption process, VAC adsorption laboratory scale unit and VAC adsorption pilot sub-unit.

B. Pre-treatment

After removing organic compounds of high value, water is treated further in order to remove suspended solids. This is done by means of coagulation, manipulating electrostatic charges of particles suspended in water, with the addition of polyaluminum-electrolytes at a specific range of pH (1st container). Settled solids are removed and clean water is regulated to a specific pH range to be further treated using an Advanced Oxidation Process (AOP).

C. Treatment of organic load

C1. Advanced Oxidation Process (AOP)

In this step, organic compounds are degraded via highly reactive, oxidative species (hydroxyl radicals), which are produced using a combination of UV irradiation (artificial UV source) and either a semiconducting catalyst (e.g. TiO_2) or hydrogen peroxide (H_2O_2), an oxidiser. This step is particularly important in case of the presence of:

1. Low biodegradability compounds as they are not effectively removed by biological treatment, or toxic materials.
2. Compounds toxic to biological treatment microorganisms.
3. Predator organisms that feed on the biological treatment microorganisms.

The aim of this step is to either totally mineralise organic compounds, or to partially break down organic compounds resistant to various degradation processes, into smaller more biodegrade species, suitable for assimilation by the next treatment step, the small bioreactor platform (SBP).

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For our purposes, we have employed two different types of photocatalytic reactors:

- A CPC photocatalytic reactor (Figure 41), which could operate using all photocatalytic systems (heterogeneous, homogeneous, slurry or immobilized catalyst etc.), under either solar or artificial light has been designed and constructed. The reactor has been tested as an individual technology under solar and artificial light using TiO_2 photocatalyst and then employed to the system.
- A closed annular photocatalytic reactor, able to include different artificial irradiation sources and chemical additives, to facilitate more effective and intensive organic degradation.

Figure 41. CS4: Photocatalytic reactor.

C2. Small Bioreactor Platform (SBP)

This module comprises of a bioreactor that will only operate if deemed necessary. The combination of AOP and SBP can lead to a significant reduction on time and cost of the water treatment process, while increasing the overall process' efficiency.

The SBP is a form of biological treatment in capsules, which means that microorganisms are encapsulated in porous material, giving them specific advantages as regards to stability, limits of operation and simplicity of application/removal.

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Figure 42. CS4: SPB capsules.

System control and operation

The process is monitored by a number of sensors measuring parameters such as pH, TSS etc and an online Total Organic Carbon (TOC) analyser. The sensors are connected to an array of controllers that are able to receive the signals from the sensors and based on simple rules can control in the process, log data, and allow remote access and monitoring (Figure 43).

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Figure 43. CS4: Pilot plant sensors, control unit and TOC analyzer.

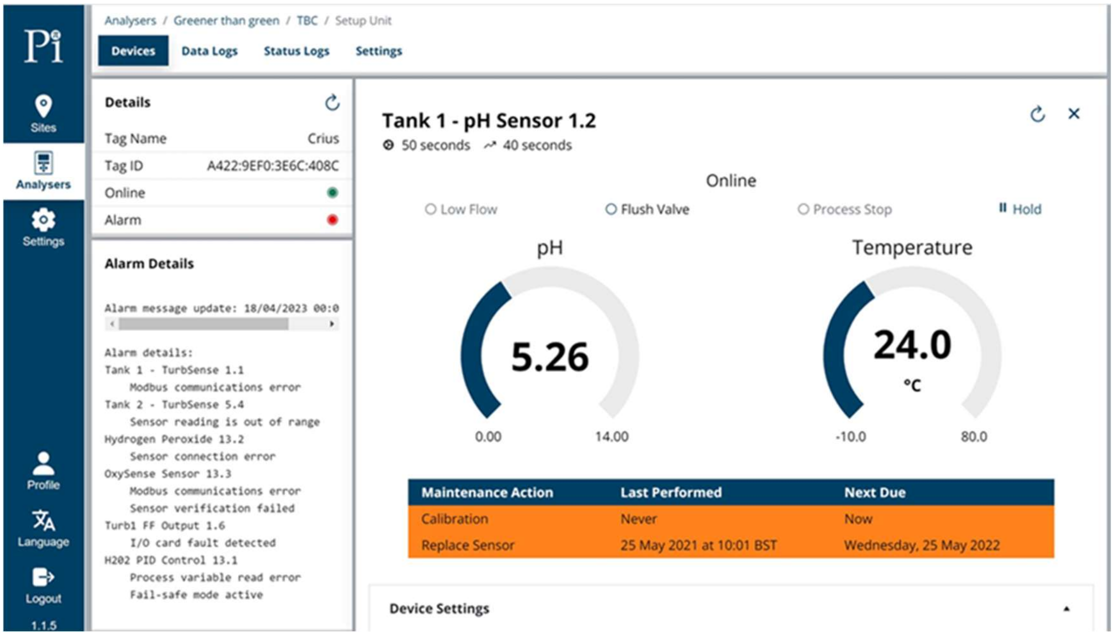

Figure 44. CS4: Remote control of the system.

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Technical requirements for its implementation and operating conditions

VesperX can operate as a standalone wastewater treatment plant or can be installed as part of an existing treatment facility. There are no extensive site requirements. Some initial testing and planning is required to determine which outflow streams are most suitable for reclaiming VAC, and to determine the remediation needs.

Within the frame of ULTIMATE, the CS4 pilot was set-up and tested/calibrated by performing control experiments.

Initial tests were performed on an abundant, frequently produced, and common stream of wastewater (Table 24). Initial pilot experiments were conducted with the following operating conditions (Table 25).

Table 24. CS4: Wastewater for start-up operation.

Wastewater TYPE 1 – Orange juice production	
TOC	2300 mg/L
pH	6.6
TSS	< 400 mg/L

Table 25. CS4: Typical operation conditions.

Operational Parameter	Value
Flow rate	10 m^3 /day
Duration of operation	8 hours/day for 5 days
Addition of NaOH / H_2SO_4	Adjustable up to pH 7
Addition of coagulant (Sodium aluminate)	0.2 to 0.7 mL/L
Addition of H_2O_2	Variable
Air sparging in SPB	0

2.4.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

In Table 26, KPI achievement can be seen.

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Table 26. CS4: Technology performance (KPI).

KPIs as specified in GA	Achievable targets - Full scale
Reduction in freshwater use by wastewater reuse (90%)	90% recovery of water (compared to the water present in industrial by-product)
Material recovery (60%)	75% total phenolic content recovered (as of total phenolics present in industrial by-product)

Recovery of high-added-value compounds (antioxidants) in Nafplio

The pilot plant operating with orange by-product stream was able to remove 80% of the available polyphenols present. The saturated resin was further treated to extract the polyphenols, which were subsequently isolated and purified. This proved to be an important step in the frame of water reuse for its contribution to step is crucial as it reduces TOC, as it presented below.

Reuse of fruit processing wastewater in Nafplio – Lab Test

Lab-scale experiments have shown that pre-treatment steps (filtration, polyphenols adsorption, pH adjustment and coagulation) achieve 50% reduction of the TOC and the remaining wastewater is colourless, odourless, and free of solids (Figure 45).

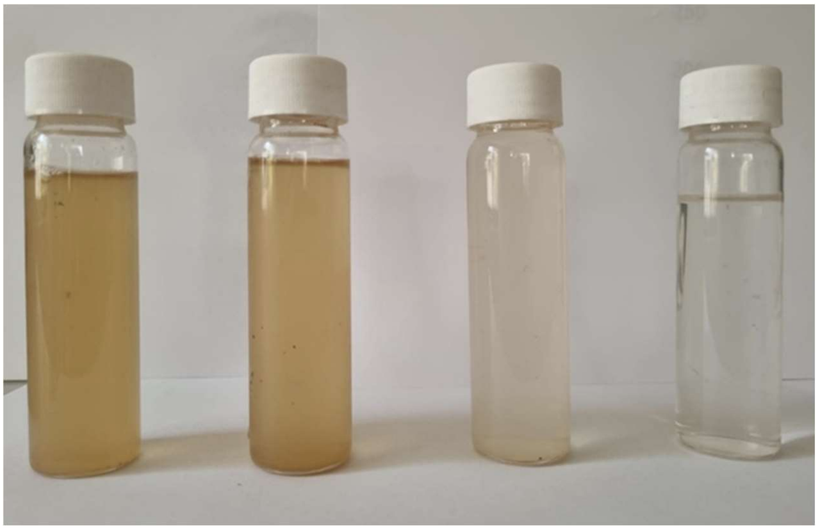

Figure 45. CS4: Pre-treatment steps of orange production line by-product.

Reuse of fruit processing wastewater in Nafplio – Pilot Plant

Adsorption

On average 80% of the polyphenols present in industrial by-product are adsorbed on the resin. This step is crucial both for the recovery of the polyphenols and for the further

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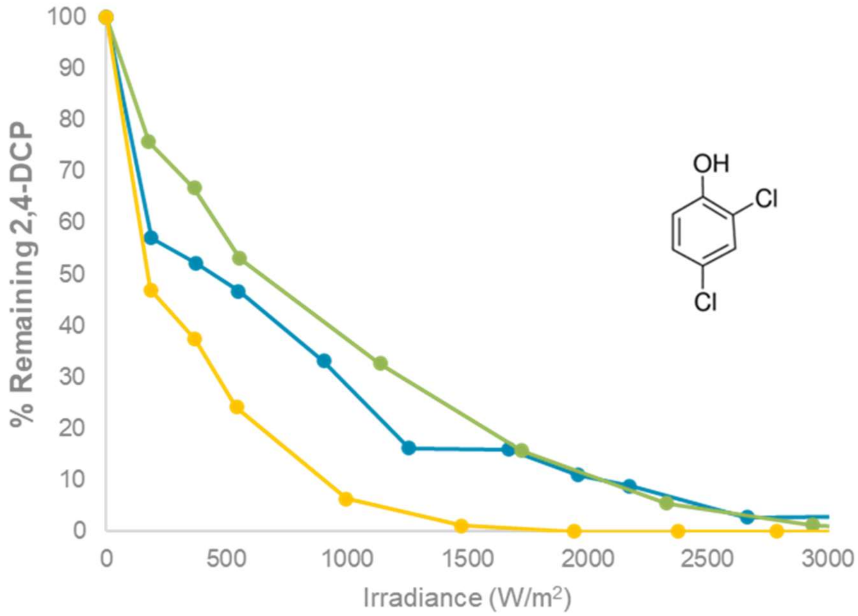
water treatment as it offers significant TOC reduction, around 20-25%. In the case of orange by-product each kg of resin can adsorb 4,7 g of polyphenols. This is quite lower than the olive wastewater where each kg of resin can adsorb 23,7 g of polyphenols present in olive oil mill wastewater. This difference is believed to arise from the presence of sugars in orange water by-product, which also have a relatively high affinity for the sorptive material surface and compete for the adsorption sites.

AOP Reactors Testing

The Solar AOP reactor was initially tested with heterogeneous catalysis and catalysts available in the market or in the experimental stage. Our results have shown that it degrades model compounds. Figure 46 presents the degradation of 2,4-Dichlorophenol and Sunset Yellow which were used as a model compounds.



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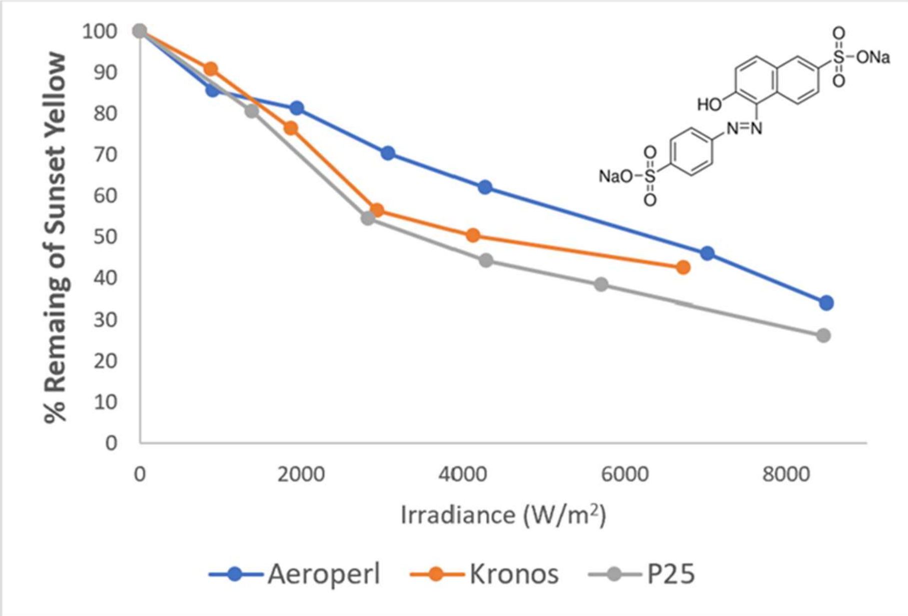

Figure 46. CS4: Degradation of a model organic compound using the CPC solar reactor.

The solar reactor was employed to degrade the orange by-product. The results in terms of TOC measurements are given in the table below:

Table 27. CS4: TOC measurements –Solar Reactor.

Irradiance W/m^2	TOC (mg/L)
0- inlet	1005
500	982
1000	1003
2000	994
3000	997
4000	1004

Although no significant reduction in TOC is observed (the organic load remains constant) it is believed that this is due to the fact that the reaction does not lead to mineralization (organic molecules degraded to CO_2) which would lead to TOC reduction. Instead, organic molecules break down into small fragments which would explain the constant TOC measurement. It was deemed that TOC analysis was not suitable for measuring the performance of this specific step, as the aim is to break the molecules into smaller ones that should be easier to metabolise by the SBP microorganisms. Consequently, TOC was used to monitor the overall performance of

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the unit, and an alternative analytical technique should be sought for monitoring this step.

With annular AOP reactor H_2O_2 was employed to avoid using catalysts in order to avoid the costly and anything but trivial separation and retrieval of the catalyst. Initially, the degradation of a model compound was tested at 6000W, with initial concentration of 1000 mg/L Sunset Yellow and 50 mg/L H_2O_2.

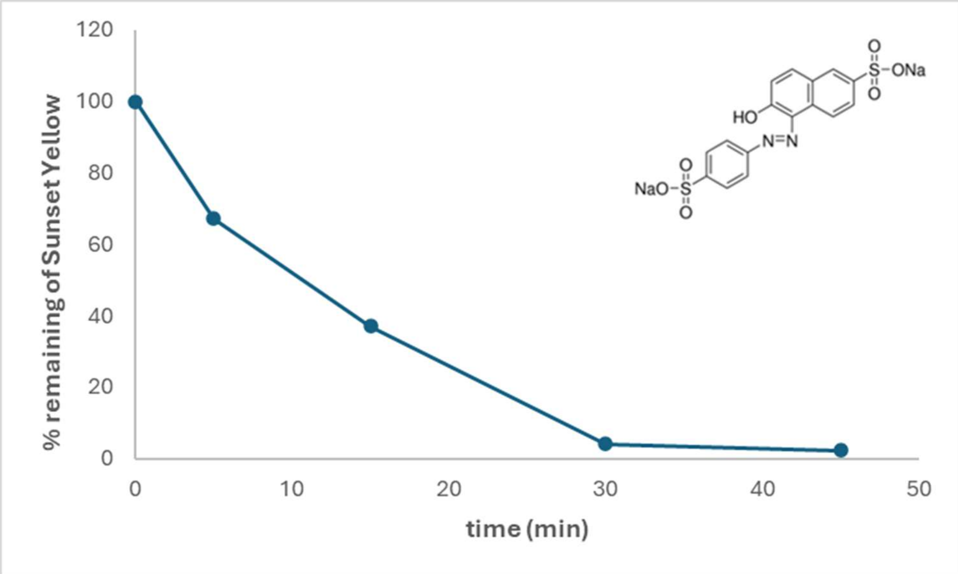

Figure 47. CS4: Degradation of a model organic compound using the annular reactor.

The model compound has been fully degraded in under 30 minutes in the annular reactor. It is worth noting that the model compound didn't not only broke down into smaller fragments but as it can be deduced from the TOC measurement approximately 50% was mineralised to CO_2.

Table 28. CS4: TOC measurements – Annular Reactor.

Time (min)	TOC (mg/L)
0- inlet	1021
5	913
15	827
30	643
45	561

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Overall results of the Case Study

In the pilot plant both with the Solar AOP reactor and the Annular Reactor were used.

Treatment prior to AOP

Larger fruit particles and other solids are removed by means of a bag filter. Adsorption significantly contributes to an approximately 20% reduction in TOC from the removal of polyphenols and to a lesser extent sugars. Subsequent coagulation removes the majority of suspended solids resulting to a relatively clear, colourless and odourless liquid that proceeds to the AOP step.

Solar Reactor Results

As previously mentioned, AOP breaks down the organic molecules into smaller ones and does not lead to mineralisation. Hence the total organic load as measured in terms of TOC remains relatively constant.

The following table illustrates the mean data from a daily operation of the unit.

Table 29. CS4: Overall mean data from a daily operation of the unit with orange stream and Solar reactor.

Step	TOC (mg/L)	Colour
Orange Wastewater	2230	Brown
Bag Filter	1870	Light Brown
Polyphenols adsorption	1460	Slightly Yellow
Coagulation	1005	Colourless
AOP	982	Colourless
SBP	NO DATA	

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Orange wastewater- Inlet **Treated wastewater- Outlet (without the SBP step)**

Figure 48. CS4: Inlet wastewater (left) and treated water (right).

Annular Reactor Results

When the annular reactor operated at 12000 W part of the organic matter was mineralised as shown in the table below.

Table 30. CS4: Overall mean data from a daily operation of the unit with orange stream and Annular reactor.

Step	TOC (mg/L)	Colour
Orange Wastewater	2420	Orange with solids
Bag Filter	1910	Light Brown
Polyphenols adsorption	1530	Slightly Yellow
Coagulation	1005	Colourless
AOP	320	Colourless

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Step	TOC (mg/L)	Colour
SBP	80	Colourless

SBP needs high volumes to operate, so we faced some issues measuring its performance. Given that the volume of the SBP tank is 250 times larger than that of the annular reactor and that the contact time of the SBP was at least 3 days, it is difficult to ascertain how each specific volume of the substance was degraded by the microorganisms.

Figure 49. CS4: Inlet orange juice by product (left), intermediate semi-treated by-product and treated water (right).

Comparison of baseline situation with ULTIMATE solution

There is no related solution in the market, pointing out the novelty of the developed technology. As baseline situation one might consider the most commonly used practice to dispose of water and the dissolved value-added compounds in a (primary) biological treatment unit, as did Alberta before the onset of ULTIMATE, a wasteful practice that sparked our interest in the matter. Characteristically, as noted in the Greenfield Assessment in WP5, the loss of value from this practice leads to a loss of value that exceeds the €1bn mark, given the assumptions of the study. Therefore, a comparison of the ULTIMATE solution with the baseline situation cannot be viewed in terms of a quantified parameter such as improvement in efficiency or % change, but in terms of a complete change in mindset viewing the water byproduct of such processes not as waste but as a recourse.

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2.4.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

Greener than Green Technologies has successfully designed constructed and optimised the VesperX prototype. Operated for the several months at the Alberta facilities, in Nafplion, Greece, the functionality of VesperX has been demonstrated in a real environment. It can treat at least 10m^3/day of water by-product originating from various juice production streams. The pilot can successfully remove and reclaim polyphenols for further purification and exploration. (as described in the material recovery report).

Regarding our initial design we made a significant change moving the adsorption step prior to any other treatment such as pH-adjustment and addition of coagulant, that irreversibly destroyed the compounds of interest. For future units it is needed to minimise the tank sizes (inventory minimisation from the operation management perspective) in order to reduce data lagging. This was our main concern in the SBP case. Furthermore, the cost benefit analysis indicated that SBP should be replaced. It is an expensive solution (0,5 € per capsule/ at least 4000 capsules needed each week) and as the annular reactor achieves partial mineralization maybe a different final step should be selected, membranes or MBR.

Another issue that needs to be addressed to make the process more cost effective is to find alternatives to the extremely high-priced adsorption material. We believe it is imperative to migrate towards more environmentally friendly and circular materials. To address these adsorption issues simultaneously we will look into natural alternatives as well as preparation/production processes. Currently, we are working on the development of a process that treats solid wastes from the same or similar industries to be suitable for adsorption the polyphenols. After one or more uses these materials can be further processed in order to render them suitable as added value animal feed. All these steps offer a new symbiotic relationship that didn't exist before, let alone another life opportunity for material considered as waste.

In terms of further product optimisation several ideas have been proposed, with the most prominent one being the reduction of the bulk of the unit, that will make it easier to transport between sites, hence both reducing the costs and environmental impact.

Finally, one of the major challenges we are facing is streamlining the process so that it is more cost effective. As we are currently targeting very high-priced value-added compounds this cost, although significant, is acceptable or even negligible. By reducing the operational costs we can target compounds of lower market price and make their recovery worthwhile. This will increase our addressable market and make our technology/product more attractive to a wider range of industries. Simultaneously, and in conjunction with our proposed business model, we will reduce the environmental

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footprint of our business, an idea and ideal deeply embedded in the company's vision and culture.

Best practices and recommendations for technology design and operation in the symbiotic frame

In order for the adopting industry to extract the maximum value out the developed technology is to build a symbiotic relationship with their stakeholders as views from the business perspective i.e. their main raw material suppliers (local and not so local farmers), water utilities and local communities. Establishing such a relationship the industry can stop competing with the local community and farmers for water recourses, most commonly extracted from wells, while the water utilities through the utilisation of the reclaimed water of a large number of industries and the utilisation of water supply networks for irrigation, (parallel to potable water networks) experience less stress in water supply during the months of high demand. Moreover, as water utilities that are in charged wastewater management benefit from significantly reduced water volumes in need of processing. Finally, the whole ecosystem benefits from the increased availability of water. It is expected that due to networking effects the gains from the adoption of VesperX will increase exponentially with the increasing number of adopting shareholders.

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2.5. Reuse of brewery wastewater for cooling water and as process water in Lleida (CS5, ES)

2.5.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

Currently, 500.000 m^3/year of wastewater are treated via conventional activated sludge in the brewery Mahou San Miguel, located in Lleida (Spain). The effluent of the brewery wastewater treatment plant is proposed to be further treated in order to produce fit-for purpose water to be reused in cooling towers. The quality to be achieved is under the framework of an existing national regulation (Royal Decree 1620/2007), which considers that reused wastewater for cooling towers has to meet the following requirements: TSS<5 mg/L; Absence of *Legionella sp.* and of *E. Coli*, Nematode eggs < 1 ut/10L, Turbidity < 1TNU. Additionally, salinity has to be removed from the wastewater for preventing scaling in cooling towers. The combination of a nanofiltration (NF) with a molecular weight cutoff of 1kDa is used as pre-treatment for the reverse osmosis (RO).

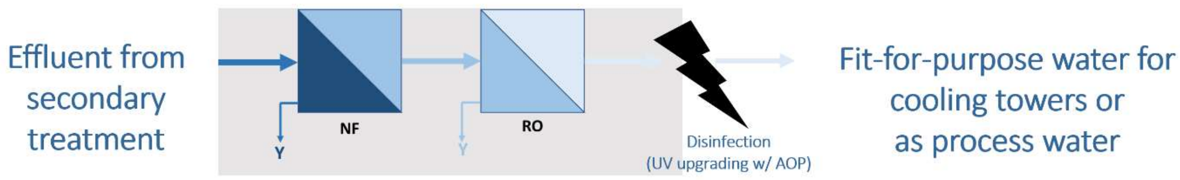

Figure 50. CS5: Process scheme.

Description of the technology, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

Nanofiltration (NF) membranes in combination with reverse osmosis (RO) membranes can be applied as a post-treatment of wastewater to produce fit-for-purpose water. It can be used to treat different water qualities such as pre-treated industrial wastewater from breweries as in the Lleida case study in ULTIMATE.

Unique selling points of the NF technology:

- Protection of RO from organic and /or colloid fouling, due to the much smaller molecular weight cut-off of the open NF membranes compared to a conventional UF
- Reduction in the number of clean in place (CIP) or membrane replacements of RO
- Reduction of the operating costs

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Although components are commercially available, no references of NF in industrial environments were found, this is why an initial TRL=7 has been considered, considering that a final TRL=9 has been achieved, once the NF+RO system has been validated.

Technical requirements for its implementation and operating conditions

Technical requirements for its implementation and operating conditions are shown in the following tables.

Table 31. CS5: Typical feed water conditions to apply the NF membrane.

Parameter	Units	Feed water requirements
Temperature	°C	0 – 40
pH	-	2 – 11
Particle size	µm	< 300
Total suspended solids	mg/L	< 200
Ozone	mg/L	0
Free chlorine	mg/L	< 500
Compounds that shall not be present in water to be treated		Organic esters, ketones, esthers, halogenated hydrocarbons, aromatic hydrocarbons, polar organic solvents

Table 32. CS5: Typical ranges for operating parameters of the novel NF membrane.

Parameter	Unit	Design by Pentair
Operation		crossflow with bleed
MWCO	kDa	1
Permeability	L/(m²*h*bar)	40-80
Flux (capacity)	L/m²/h (m³/h)	20-40 (0.8-1.6)
Recovery	%	60-80
Cross flow velocity	m/s	0.2 – 0.4
System pressure	bar	max. 6
TMP	bar	0.2 – 1 (max. 6)
Chlorine resistance	ppm hour	100 000

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2.5.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

The KPI “Reduction of fresh water through re-use of treated wastewater = 10%” has been reached. Water consumption in brewery is ca. 1400 m^3/d, and water reuse potential (only for the studied cooling towers use) can be as high as >15% of daily needs, which could potentially be provided by the proposed solution.

The key performance indicators are shown in the following table.

Table 33. CS5: Key performance indicators for the novel NF membrane in CS5.

Parameter	Unit	Value obtained in CS5	Value extracted from the estimation by Pentair
Turbidity Permeate	NTU	< 0.4	< 0.2
Total suspended solids Permeate	mg/L	< 3	< 0.2
Bacteria (*E. coli*)	Log removal	> 5	> 4 – 6
Viruses (som. Coliphage)	Log removal	n.d.	>2 – 4
Parasites (*C. Perfringens*)	Log removal	n.d.	>4 – 6
Intervals backwash of NF	min	60	30 – 60
Intervals of CEB/CEC of NF	h	≥ 24	24
Intervals of CIP of NF	months	n.d.	3 – 12
Intervals CIP of subsequent RO	weeks	n.d.	1 – 2

CEB – chemical enhanced backwash

CEC – chemical enhanced cleaning

CIP – cleaning in place

n.d. – not determined

NF could operate without coagulant (which means savings in terms of operational costs), obtaining a reasonable flux through the membranes (24 L/m^2/h), working at a minimum crossflow velocity (0.2 m/s) and a high recovery (75%).

Up to 400 m^3 of reusable blended water was supplied to the brewery’s cooling towers during all the action and were satisfactorily reused by the brewery.

Two plug-and-play demo plants (one for NF, the other for RO) were installed in the WWTP of Mahou San Miguel (see next figure).

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NF plant was supplied by Pentair, known as NanoFlex Pilot 80-81 and it includes one $40m^2$ NF module, model HFW1000.

RO plant was supplied by Aqualia, and includes two standard 8" RO modules.

Nanofiltration demo plant

Reverse osmosis demo plant

Figure 51. CS5: Nanofiltration and reverse osmosis pilot plant.

Both plants were installed by Aqualia ending 2021. Between January and August 2022 both plants were operated. During this period Pentair and Aqualia had weekly follow-up meetings for monitoring of the solution. Up to 25 samples from each stream (feed (treated wastewater), NF outlet and RO outlet) were taken ad analysed during the abovementioned period. Additionally, 10 samples of remineralised water (RO outlet + NF outlet) were taken for validating the produced reusable water.

Comparison of baseline situation with ULTIMATE solution

This experience took place in a local drought context, where reusing water is a known, reasonable and affordable option. The experience was well welcome by the brewery. Aqualia elaborated a basic engineering project to a deep understanding of the upscaling of the NF+RO train. Aqualia is currently still under conversations with the brewery, who is looking for implementation of water saving strategies.

2.5.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

- NF is a valid technology for meeting water reuse regulatory requirements, but for salinity removal a further RO step is needed. Then, a blending step is necessary, to decrease corrosion.
- 800Da is enough membrane cut-off for removal of pollutants.
- Conversion should be kept as low as possible to optimize filtration performance.
- NF must be well case-based justified, since NF is less competitive in CAPEX and OPEX than conventional UF.

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- Pre- or post-treatment can be required, depending on the water characteristics.

Best practices and recommendations for technology design and operation in the symbiotic frame

Pilot trials are essential for warranting a successful industrial-scale business case. Pre- or post-treatment can be required, depending on the water characteristics. The implication and willingness of the end-user (like the brewery in this case) is essential. Environmental benefits of water reuse are clear, but economic benefits might not be, due to low water price in dry countries like Spain. Public incentives / private strategies for water reuse or drought scenarios can be key drivers.

Crucial factors for technology implementation and its optimal performance

- Policy and regulation in water reuse is key. A coordinated elaboration between administrations and users, water reuse incentives, demonstration campaigns of the water reuse capacity / potential and authorization of indirect or direct potable reuse will contribute on water reuse.

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2.6. RO treatment of distillery wastewater after AnMBR for internal water reuse in Tain (CS7, UK)

2.6.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

Case Study 7 is the Glenmorangie whisky distillery located in Tain, in the north-east of Scotland. The current effluent treatment plant, at the start of the symbiosis between the distillery, the water sector and farmers, was designed and installed by Aquabio, partner in the ULTIMATE project, and consists of screens followed by an anaerobic membrane bioreactor (AnMBR) to treat the wastewater generated during the whisky making processes. The treated effluent is then discharged in the local estuary, the Dornoch Firth. The main target for the current treatment train is to remove the organic content, achieving over 98% removal of the 10.7 t/d COD load. In the AnMBR, the COD is biodegraded and converted to a methane rich biogas which is processed in a boiler to produce heat currently used for heating the stills in the distillery. Overall, this reduces the dependence in fossil fuels of the distillery by 15%. In addition, the excess sludge produced in the AnMBR is provided to the local farmers for application to the fields as soil enhancer.

It should however be noted that the effluent for discharge still contains concentrations of ammonium and phosphorus of about 700 mgN/L and 250 mgP/L, respectively, offering potential for recovery as fertiliser, for example. Also, the AnMBR is operated in the mesophilic range; thus, its effluent has a temperature between 35 and 40°C which provides an opportunity for residual heat utilisation within the treatment facilities, reducing the overall energy demands of the additional technologies being considered. Finally, the effluent can be further treated to produce high quality water that can then be reused at the distillery for cleaning or cooling purposes. This would then lead to a significant reduction in tap water demand for these uses. As part of the ULTIMATE project, Aquabio and Cranfield University (partners in the project) are collaborating with the Glenmorangie distillery and Alpheus, the current operator of the treatment site, to evaluate options to expand the circular economy approach at distilleries with residual heat utilisation, nutrients recovery and water recycling.

The distillery industry is known to have a very high-water consumption for both the main production and other processes such as cooling and cleaning. While the main production usually uses natural waters, tap water tends to be used for the other applications. In our case study, the distillery uses approximately 1800 m^3 per week of spring water for production only and another 1690 m^3 per week of tap water for the cooling towers, heat exchangers and cleaning. This represents an operational expenditure of about 92 k€/year. Interestingly, the wastewater flow rate produced is about 322 m^3/d which is higher than the tap water usage due to also some contribution

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of the spring water from the production. This then shows that there is a great potential to reduce freshwater consumption by treating and reusing the wastewater. From a perception point of view, distilleries are very unlikely to use recycled water for the production of the whisky so the opportunity for water recycling is only sources for the tap water uses. As stated above, the current AnMBR effluent still contains high levels of nitrogen and phosphorus as well as some organics and salinity (Table 34). Comparison to water quality standards for cooling water, as an example, highlights that significant treatment is still required to produce high quality water for reuse (Table 34). As part of this project, a treatment sequence comprising nutrients removal and recovery technologies with struvite precipitation and ammonia stripping combined to reverse osmosis (RO) membranes to furthers remove other contaminants and salinity was studied. Due to their ability to remove salts from water RO membranes have largely been applied for seawater desalination but they are also applied for the purification of treated effluents for water reuse, generally where high quality is required such as potable uses. RO membranes can achieve near complete removal of salts and other contaminants from water such as viruses, ions and organics. Typically, RO membranes are thin film composite membranes with a polyamide active layer and configured in spiral wound elements. The water to be treated is fed under pressure in the membrane elements producing a clean permeate at one end and a concentrate (brine) on the other.

Table 34. CS7: Characteristics of the AnMBR effluent and example of water quality criteria for cooling.

Parameters	AnMBR effluent	Standard for cooling water
pH	6.91	6.9-9.0
EC (mS/cm)	6.35	-
COD (mg/L)	805	75
TN (mg/L)	640	-
TAN (mg/L)	676	1
PO_4-P (mg/L)	221.5	4
Cl^- (mg/L)	209	500
SO_4^{2-} (mg/L)	1138	200
Ca^{2+} (mg/L)	92	50
Mg^{2+} (mg/L)	68	-

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Parameters	AnMBR effluent	Standard for cooling water
K^+ (mg/L)	669	-
Na^+ (mg/L)	349	200

Description of the technology, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

As RO is an anergy intensive process and its performance can be significantly affected by membrane fouling (deposition of contaminants on the surface of the membrane), pre-treatment is often critical to ensure smooth operation. However, in the current context, using the RO membranes capability to concentrate pollutants may be beneficial for the removal and recovery of the nutrients. Indeed, it has been reported that efficiency of nutrients recovery systems is improved with concentrated streams. Consequently, as part of this work, we aim to first and foremost demonstrate the potential of RO membranes to produce high quality water from distillery wastewater for reuse but also evaluate the potential to adapt the sequence in which the nutrient recovery technologies and the RO membranes are implemented to optimize the sustainability of the treatment train. For this, three scenarios were evaluated (Figure 52). In the first one (sequence 1), the RO membranes were placed first, just after the AnMBR. The expected advantage here is to produce a smaller volume of concentrate with the nutrients then leading a reduce investment for the nutrient recovery technology due to the reduced flow to be treated. The expected disadvantage is that the RO membranes will be exposed to higher levels of contaminants leading to potentially more fouling and possibly also a poorer permeate quality for reuse. Sequence 2 then looked to implement first the struvite precipitation to remove key scaling agents such phosphorus, magnesium and calcium (likely to precipitate at the same time) to protect the membranes (i.e. reduce fouling) but still apply the ammonia stripping in the concentrate stream from the RO to reduce flow and improve efficiency. Finally, in Sequence 3, both nutrients recovery technologies were implemented before the RO membranes. In this case, the aim is to fully protect the RO membranes by sending a cleaner stream as feed leading in principle to better operation and permeate quality, but all technologies will have to treat the full flow so leading to a higher investment cost for larger systems. There are then trade-offs to be explored between these different scenarios.

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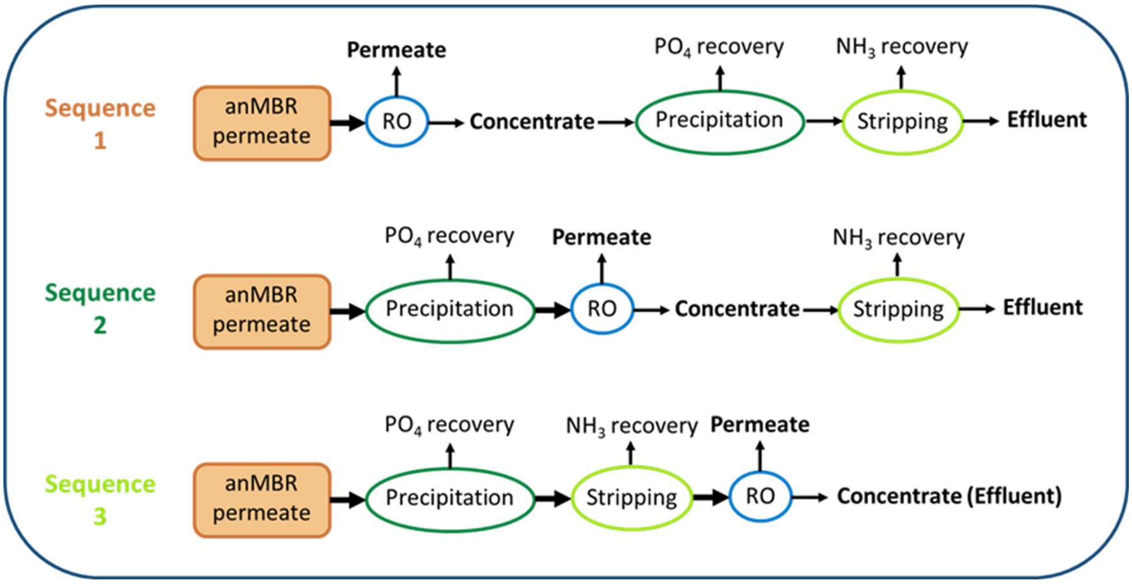

Figure 52. CS7: Scheme of the three scenarios.

Figure 53. CS7: Photos of the struvite reactor (left) and ammonia stripping units (right).

Technical requirements for its implementation and operating conditions

The work was carried out at a combination of lab and pilot scales. For practical reasons the nutrients recovery steps were performed at lab-scale while the RO membranes, the focus of this task, were tested at pilot scale (Figure 54) with thin-film composite spiral wound membranes (TriSep, TurboClean 1812-X20-31). The feed pressure was fixed at 16 bars and the feed flow was 0.1 L/s. The system was operated in semi-continuous conditions where the feed was recirculated to achieve a set recovery similar to full scale systems. The recovery, conversion rate of feed into permeate, was 50% unless specified otherwise. According to the 3 sequences tested, the RO pilot plant

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was fed directly with raw AnMBR effluent for sequence 1 (high N and P), precipitated AnMBR effluent for sequence 2 (high N, low P), precipitated and stripped AnMBR effluent for sequence 3 (low N and P). For each source, the pH was adjusted to below 7 with a H_2SO_4 solution as typically applied in full systems to control scaling.

For the struvite precipitation, the trials were carried out with a multiple jar tester (Phipps & Bird's), with a sequence of fast mixing for 5 minutes at 150 rpm during which magnesium chloride was dosed to obtain a P:Mg ratio of 1:1.3 and the pH was adjusted to 8.3 with caustic. Mixing was then reduced to 60 rpm for 55 minutes to allow for the precipitation reaction to occur. The mixture was then left to settle for 30 minutes to sperate the precipitate from the water and subsequently filtered at 5 µm before use for further testing with the RO membranes and for analyses.

Ammonia stripping was performed using a rotary evaporator (BUCHI's Rotavapor R-100) for thermal vacuum stripping. In these trials, the pH was adjusted to 10.5 with caustic so that most ammonium was converted to the volatile form, ammonia. The trials were then carried out at a temperature of 67°C and vacuum pressure of about 200 mbar.

Figure 54. CS7: RO membranes pilot plant.

The technologies evaluated here are already commercially available but their application for distillery wastewater had, to our knowledge, not yet been done with also no reports of their integration for such industrial applications. Based on this, the systems were at a TRL of 5 at the start of the project and through the trials carried out here with real wastewater that increased to TRLs of 7 respectively. This work also looks to expend the existing symbiosis between the distillery and the water sector through the involvement of technology providers and operator for the potential treatment train and the also the local water utility (Scottish Water in this case) with a possible change in freshwater demand. Although the water is to be reused directly at the distillery, implementing water recycling on site would indeed lead to a reduction in potable water usage and overall help the water utility with water resources management (potential reduction in freshwater abstraction) locally.

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2.6.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

The characteristics of the influent and permeate of the RO membranes for each sequence tested are presented in Table 35. As stated before, the expected key in the different feeds were low phosphorus levels in sequence 2 and 3 with 5.7 and 7.2 mgP/L, respectively, when compared to 222 mgP/L in the AnMBR effluent, low levels of ammonia (TAN) in sequence 3 with 70.4 mgN/L as opposed to over 600 mgN/L in the other two. Interestingly, the removal efficiencies of the nutrients recovery systems at lab scale with over 97% for the phosphorus and 90% for the ammonia were found to be higher than those reported in the demonstration units tested as part of this project (see D1.5 for details). This highlights that further improvement can be achieved in the demonstration systems and also, to some extent, provide an improved scenario for the RO membranes performance studied here. It is important to note that other differences can be found in the influents for the 3 sequences. Indeed, the electrical conductivity (EC) increased from 6.35 mS/cm in sequence 1 to 8.49 and then 10.5 mS/cm in sequences 2 and 3. Similar increases can also be observed for the sulphate, chloride and sodium ions. These differences can be explained by the chemical dosing needed for pH adjustment and magnesium addition for the different processes which will increase the concentration of specific ions and hence the overall salinity of the source water to be treated. For precipitation the pH was increased to 8.3 with sodium hydroxide, magnesium chloride was dose for the struvite formation and the pH was adjusted back down to below 7 with sulphuric acid for the RO trials, explaining the increased concentrations in chloride, sodium and sulphate and the overall salinity. For the stripping the pH was increased up to 10.5 and again decreased back down for the RO trials explaining the even higher levels measured. Generally, this highlights a limitation in processing the water in the nutrients recovery technologies before the RO (sequence 2 and 3) as the increase salinity leads to a higher osmotic pressure hence a requirement for a higher feed pressure in the RO to achieve a set flux and more energy use. However, as expected, the RO membrane delivered excellent removal of all contaminants in all sequences tested. To illustrate, the salinity (as conductivity) was reduced by more than 95%, the organics (as COD) by 99%, the phosphorus also by 99% and other ions by at least 96%. Due to the concentration-polarisation phenomenon occurring at the surface of the membrane and the fact that passage of compounds through RO membranes occurs through diffusion, increased concentrations in the feed will generally lead to higher concentrations in the permeate as observed for the most compounds reported here. However, due to the excellent performance of the RO membranes all parameters were well below the criteria required for cooling water (Table 34) except ammonia. The criteria for ammonia for cooling water is set at 1 mgN/L whereas, at best, an average concentration of 3.3 mgN/L was achieved in the permeate in sequence 3. Because of that relationship between feed and permeate concentration, this could be improved by further improving the

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performance of the ammonia removal and recovery technologies before the RO. An alternative approach is to further treat the permeate produced. This can be done through a two pass RO system in which the permeate from a first set of RO membranes is filtered again through a second set. In the current work, this was tested based on sequence 1 (*Table 36*). For these trials, different recovery rates from 50 to 85% were tested. A second RO pass of course reduced all the contaminants to very low levels in the final permeate. For the ammonia, concentrations of below 1 mgN/L, the target for the cooling water, were achieved for recoveries of 50 to 80%. When the recovery was increased further to 85%, the ammonia concentration moved just above that 1 mgN/L limit. According to the same processes described above (concentration-polarisation and diffusion), an increase in recovery leads to a significant increase in concentration on the feed side and ultimately a slight increase in concentration in the permeate. These results show that it is possible to obtain a very high-quality water with a two-pass RO system but depending on the initial concentration of, in particular, ammonia, maximum recoveries may be limited. It should also be noted that not all applications may have such strict requirements for ammonia and single pass RO will be suitable (e.g. cleaning).

Table 35. CS7: Characteristics of the influent and permeate from the RO membranes for the different sequences tested.

	Sequence 1		Sequence 2		Sequence 3	
Parameters	**Influent**	**Permeate**	**Influent**	**Permeate**	**Influent**	**Permeate**
pH	6.91	8.78 ± 0.52	6.62	7.99 ± 0.29	6.68	7.77 ± 0.05
EC (mS/cm)	6.35	0.29 ± 0.01	8.49	0.21 ± 0.01	10.5	0.12 ± 0.00
COD (mg/L)	805	5[1]	556	5[1]	669	6.90 ± 0.70
TN (mg/L)	640	30.3 ± 5.0	620	13.0 ± 1.7	83	10.0[1]
TAN (mg/L)	676	37.0 ± 5.3	602	20.6 ± 1.6	70.4	3.3 ± 0.1
PO_4-P (mg/L)	222	0.11 ± 0.02	5.7	0.05[1]	7.2	0.05[1]
Cl^- (mg/L)	209	4.2 ± 0.9	641	12.9 ± 1.4	706	9.80 ± 0.1
SO_4^{2-} (mg/L)	1138	0.5 ± 0.1	2721	1.40 ± 0.03	4315	3.01 ± 0.04
Ca^{2+} (mg/L)	92	2.90 ± 0.84	138	2.17 ± 0.28	131	2.22 ± 0.20
Mg^{2+} (mg/L)	68	0.79 ± 0.30	184	1.27 ± 0.24	188	1.01 ± 0.17

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	Sequence 1		Sequence 2		Sequence 3	
Parameters	Influent	Permeate	Influent	Permeate	Influent	Permeate
K^+ (mg/L)	669	12.83 ± 1.16	659	9.04 ± 0.67	704	5.67 ± 0.03
Na^+ (mg/L)	349	6.08 ± 0.27	884	8.78 ± 0.36	2300	19.47 ± 0.14

[1]: concentrations below the detection limit

Table 36. CS7: Characteristics of the permeates in a two pass RO system based on sequence 1.

Parameters	Permeate 1st pass	Permeate 2nd pass			
		50%	75%	80%	85%
pH	6.61	9.11	9.66	9.92	9.92
EC (µS/cm)	178.3	12.14	19.84	23.3	27.2
COD (mg/L)	5[1]	5[1]	5[1]	5[1]	5[1]
TN (mg/L)	14	10[1]	10[1]	10[1]	10[1]
TAN (mg/L)	16.8	0.2[1]	0.32	0.57	1.14
PO4-P (mg/L)	0.26	0.05[1]	0.05[1]	0.05[1]	0.05[1]
Cl^- (mg/L)	5.68	0.53	0.30	0.42	0.49
$SO4^{2-}$ (mg/L)	7.69	0.45	3.66	0.04	1.77
Ca^{2+} (mg/L)	3.03	3.13	0.93	0.85	1.22
Mg^{2+} (mg/L)	1.40	0.13	0.25	0.04	0.10
K^+ (mg/L)	11.33	0.72	1.14	1.51	1.78
Na^+ (mg/L)	4.88	0.25	0.40	0.57	0.57

[1]: concentrations below the detection limit

The characteristics of the wastewater to be treated can influence operational conditions in an RO system due to the salinity and hence the osmotic pressure and the fouling rate due to the interactions of the contaminants present with the membrane. A highlighted by the change in salinity in the feed for the different sequences studied

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the estimated osmotic pressure increased from about 3.6 to above 5 bar between sequences 1 to 3 (Figure 55). This means that at a set system pressure, as it was the case in these trials, the net effective pressure will be reduced (Pnet = Papplied-Posmotic). The net pressures were then 12.4, 11.8 and 10.8 bar for sequence 1, 2 and 3, respectively, leading to lower fluxes in sequence 2 and 3. Alternatively, in a full-scale system, a higher pressure would have to be applied to maintain set flux resulting in higher energy cost. Normalising the data obtained from the current trials into permeability ($k = flux/P_{net}$) showed that a higher value was obtained for sequence 1 than for sequence 2 and 3. This, however, goes against the initial expectation that more fouling, represented by a lower permeability, would form in sequence 1 as the membrane was generally exposed to higher levels of nutrients which can contribute to scaling. This then shows that the increase in salts due to the pH adjustments not only had an impact on the osmotic pressure but also on the fouling propensity of the water. This is further evidence that sequence 2 and 3 would lead to more complex and costly operation of the RO membranes. A mass balance of the different contaminants measured (difference between the feed and the permeate and concentrate) suggests a significant accumulation of the chloride, sodium and sulphate on the membrane further demonstrating the increased risk of scaling in sequence 2 and 3 (Figure 56). Additional sacrificial tests were then conducted in a bench-scale dead-end filtration cell to further evaluate fouling and the results pointed to the formation of struvite crystals on the membrane in sequence (Figure 57). Struvite is known to naturally form in this wastewater which could lead to scaling if not controlled through antiscalant dosing or by implementing the struvite precipitation step before the RO. Overall, trials over longer term and at larger scale will be required to fully assess the fouling potential and control requirements.

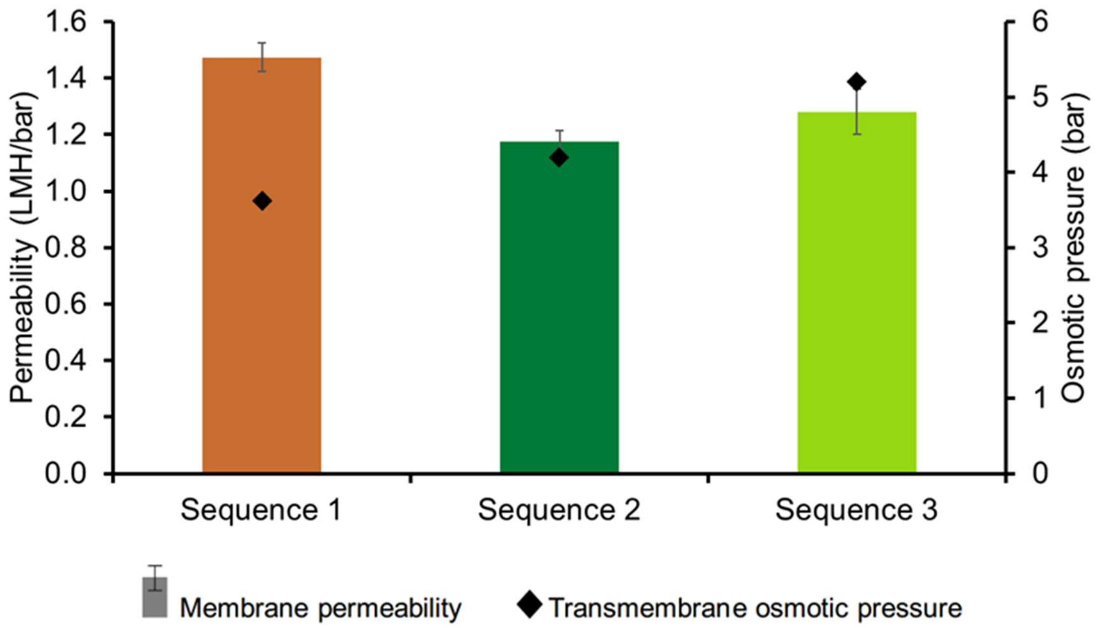

Figure 55. CS7: Osmotic pressure and permeability of the three sequences tested.

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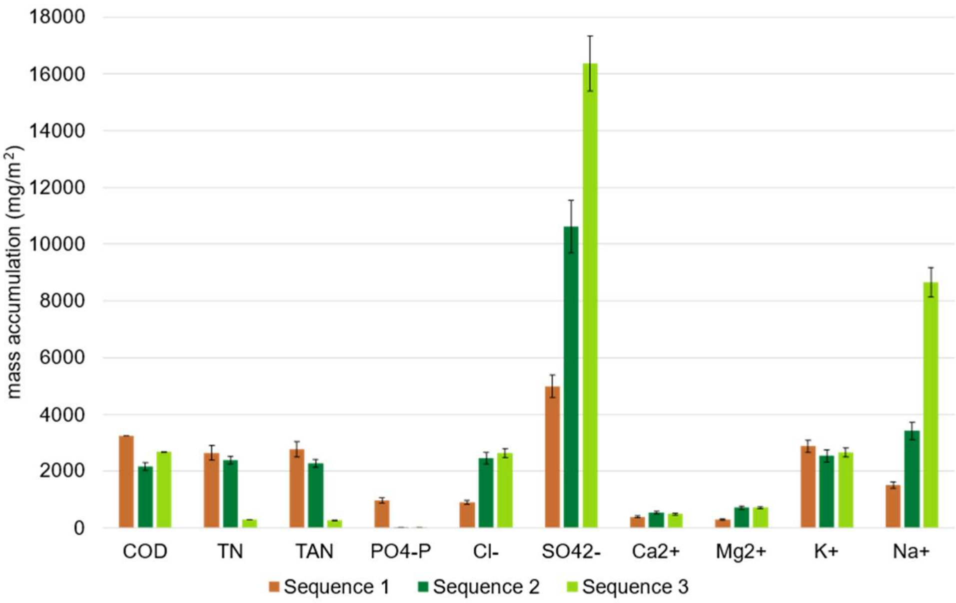
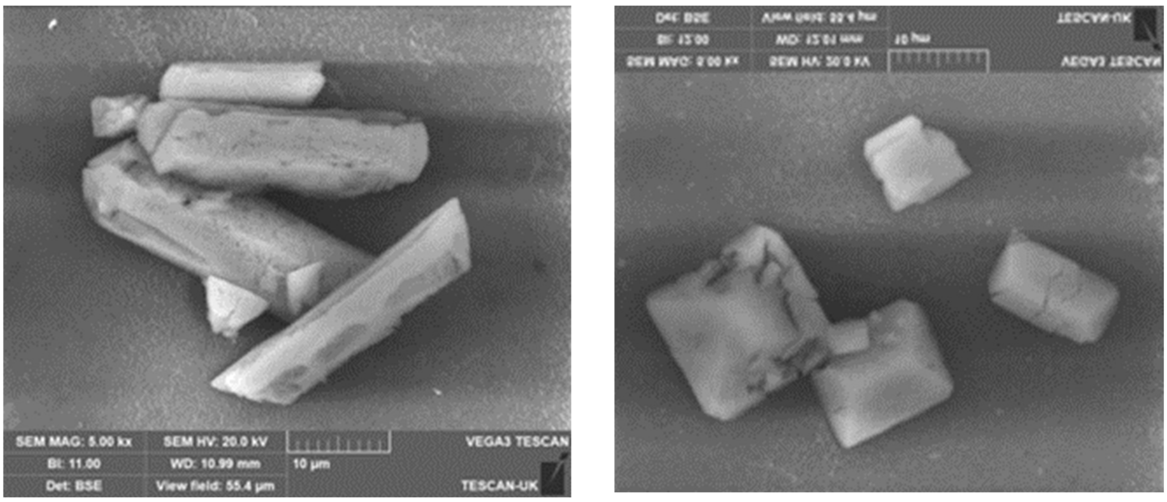


Figure 56. CS7: Mass accumulation of contaminants on the membranes.



(a) (b)

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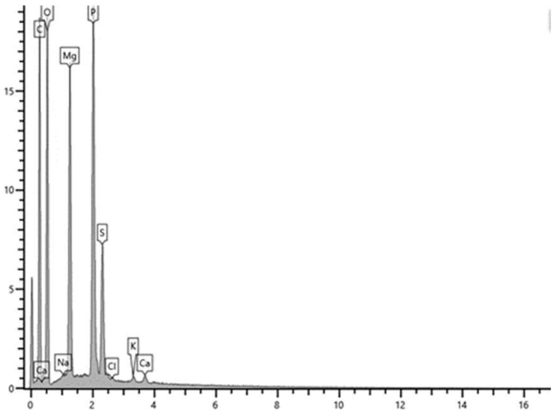

(c)

Figure 57. CS7: Scanning electron microscope images (a and b) and energy dispersive spectroscopy analysis (c) of crystals observed on membrane surface after sacrificial sequence 1 replicate trials at bench scale.

Comparison of baseline situation with ULTIMATE solution

As part of the project, the KPI was a reduction of freshwater consumption through reuse of the treated wastewater of over 40%. At full scale, assuming a 50% recovery from the 322 m^3/d of wastewater, the recovered water could in fact cover up to 66% of the tap water consumption, a very significant reduction when compared to the current situation.

According to reported typical capital expenditure for RO membranes for seawater desalination, usually based on a two-pass configuration, of between 1,000 and 2,500 €/(m^3/d of flow treated), an investment cost of about 300 to 750 k€ can be expected. Similarly, the operation expenditure has been reported at about 0.5 to 1 €/m^3 then corresponding to about 55 to 110 k€/year. Considering that the distillery wastewater considered here has a significantly lower salinity than seawater, the energy cost is expected to be lower than for desalination. Also, as the two-pass configuration may not be needed depending on the quality required, the true cost is more likely to be at the lower end of these ranges. As stated before, the cost of the current tap water usage is around 92 k€/year so if the recovery is optimised, there is potential to make such reuse scheme financially sustainable. As a way of illustrating this, if the 92 k€/year saving is invested in the reuse system with 55 k€/year operation expenditure, there would be a difference of 37 k€/year to be invested towards the capital expenditure then leading to an estimated payback period of about 8 years for an installation costing 300 k€.

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2.6.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

The current work demonstrated that distillery wastewater can be treated to high quality water for reuse. In our case study, the treatment train integrated an anaerobic membrane bioreactor and reverse osmosis membranes. The fact that an AnMBR, with ultra-filtration membranes, was used means that the effluent fed to the RO membranes was essentially solids free which is critical to avoid any clogging in the channels of the RO membranes. For implementation on other sites, where other biological processes may be in place without the membrane separation, then micro or ultra-filtration of the effluent from the biological process will be required (or at least some cartridge filters).

Due to the broader scope of this case study, not only looking at water reuse but also at nutrients recovery from the distillery wastewater, these systems were also tested for further integration with struvite precipitation and ammonia stripping for nutrients recovery. Implementing the nutrients recovery systems between the AnMBR and the RO highlighted a key limitation with increased pressure requirements and fouling propensity. Indeed, due to the need for increased pHs for the reactions to occur in both nutrients recovery systems, the salinity of the water was increased. Although, the levels of nutrients were significantly reduced through these systems, which in turn should reduce the fouling propensity of the water into the RO, the increase in salts led to more fouling than direct filtration of the anaerobically treated effluent. Interestingly, the results obtained suggest that application of the RO membranes directly after the AnMBR then followed by implementation of the nutrients recovery technologies in the concentrate line maybe more economically viable. The initial results obtained here suggested a lower energy demand of the RO membranes and the capital expenditure of the nutrients recovery systems will be reduced as they will only be treating a fraction of the initial flow. It should however be noted that if the reuse application requires very high quality, a two-pass RO system may be required. This will affect the investment cost for the RO and a more detailed economic assessment will be required to assess the viability of these options. When a single pass RO system is suitable, a two-stage process may be applied to maximise the recovery (not evaluated here). In this case, the second set of RO membranes is applied to further treat the concentrate stream and recover more of the water but again this will affect the economics of the treatment with also higher pressure and hence energy required. Overall, the work delivered in this case study showed that water reuse from distillery wastewater is achievable from a technological standpoint but there are trade-offs between performance and economics that need to be considered when defining the treatment train to be applied.

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Best practices and recommendations for technology design and operation in the symbiotic frame

Crucial factors for technology implementation and its optimal performance
It is critical that a solids free effluent is fed to the RO membranes so an MBR (as it is the case here) or implementation of UF or MF membranes as pre-treatment may be required Minimising the increase in salinity due to pH adjustment in the previous steps will ensure more sustainable operation of the RO membrane system The feed water characteristics with in particular monovalent ions such as ammonia will dictate the performance of the RO membrane system. A two-pass configuration may be required to achieve greater removals to meet the stricter consents for reuse.

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2.7. Novel membrane treatment for biotech or biotech + municipal WWTP effluent for water reuse in Kalundborg (CS9, DK)

2.7.1. Case study and ULTIMATE concept

Description of the demo site and its symbiosis cluster incl. goal

The Kalundborg Industrial Symbiosis Association exists since 1972 and interlinks seventeen private and public companies. The local industrial sector includes petrochemical, light building construction material, food, pharma, biotech, energy and bioenergy as well as waste processing. Different circular economy approaches for water, energy and materials are already implemented, e.g. the reuse of cooling water for steam production, the reuse of gypsum from exhaust gas cleaning to produce plasterboards, integrated heat management and the transfer between the industries and the district heating network as well as heat recovery from process water for district heating.

The Kalundborg Industrial Symbiosis is currently expanding. As a result, water demand is expected to double in the next two years and triple in the next five years. As the extraction of water from groundwater of drinking water quality is limited, the water extraction from the lake may have to be reduced in the near future and longer drought periods are expected, the water levels in the groundwater and the lake are very likely to decrease, if the business as usual does not change. Recent developments are therefore forcing all stakeholders to look for alternative sources of water. One possible water source is further treated effluent from the mWWTP as fit-for-purpose water.

Description of the concept, (unique) selling points of the technology/concept, TRL, capacity of technology, point of application

The mWWTP Kalundborg treats different wastewaters: municipal wastewater, pre-treated industrial wastewater and wastewater resulting from a nearby power plant. Due to the high fraction of industrial wastewater (50%), the electrical conductivity, the concentrations of organic matter (TOC, COD), calcium, hydrogen carbonate and sulfate were double to four-fold higher than for typical municipal wastewater (Levlin 2007; Henze and Comeau 2008; Ho et al. 2023). The higher organic matter concentrations, coming from the pre-treated industrial wastewater, indicated that this fraction was non-degradable in the mWWTP. The high concentrations very likely lead to organic fouling and scaling on the RO membranes, and hence, increase the operational transmembrane pressure. In addition, the electrical conductivity gave also a hint to a high salt content and together with the high content of total suspended solids (TSS), an increased pressure for the RO operation was expected. The increased pressure was expected to lead to a higher energy consumption of the ROs and to

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require a higher frequency of cleaning procedures, increasing the operational costs. In Table 37, the effluent quality from the secondary clarifier of the mWWTP is shown.

Table 37. CS9: Composition of the outlet of the secondary clarifier of the municipal WWTP. TOC: total organic carbon; COD: chemical oxygen demand.

Parameter	Unit	Content	n
Electrical conductivity	µS/cm	2300 – 6200	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 – 1300	26
Sulphate	mg/L	280 – 610	27

In Kalundborg, fit-for-purpose water for industrial reuse such as cooling, or stream production should be produced. Furthermore, the feasibility of using the fit-for-purpose water for cleaning trucks or streets will be investigated. A typical treatment train for the production of such water is the combination of ultrafiltration (UF) or nanofiltration (NF) as pre-treatment for reverse osmosis (RO). These treatment trains were tested for the effluent from the secondary clarifiers of Kalundborg WWTP.

In two parallel operated pilot plants the following membranes were tested: a conventional ultrafiltration membrane (UF) with a molecular weight cut-off (MWCO) of 150 kDa, a novel ultra-tight UF (u-t UF) membrane with an MWCO of 4 kDa, a conventional open nanofiltration membrane (NF) with a MWCO of 1 kDa followed by conventional reverse osmosis membranes. As an additional step before the UF/NF a continuously operated Dynasand filter and a dual media filter were tested. A flow scheme is shown in Figure 58 and pictures from inside the pilots are shown in Figure 59.

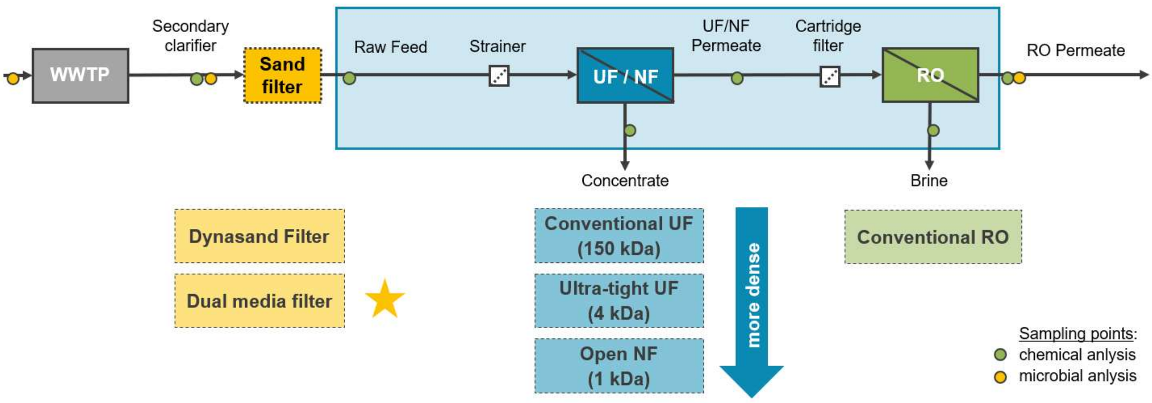

Figure 58. CS9: Pilot plant – flow scheme, tested pre-treatment and membranes and sampling points.

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The novel ultra-tight UF membrane was developed using the Layer-by-Layer (LbL) technology and consists of hollow fibers with a diameter of 0.8 mm and filtrates from inside out. The materials of the fibers are polyvinylpyrrolidone and polyethersulfone (Pentair 2022). A module has a diameter of 0.2 m and is 1.5 m long, with 12.000 membrane fibers and a membrane area of 40 m² (Figure 60). Technical details of the tested membranes can be found in Table 38.

Figure 59. CS9: View inside the pilot plant: left: pilot container A (UF/NF); middle: pilot container B (UF/NF); right: pilot container B (RO).

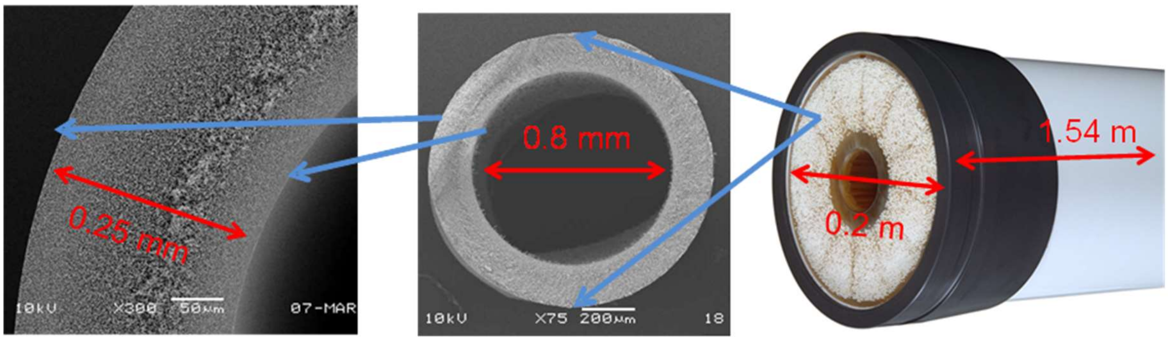

Figure 60. CS9: UF/NF membrane with microscopic details and dimensions of membrane fibres; left: fibre wall, middle: fibre, right: module head.

UF and NF membranes are hollow fiber membranes and can be backwashed with water or chemicals against the filtration direction. Also, chlorine can be used as cleaning agent (below limits of manufacturer, according Table 39).

The RO is a spiral wound membrane, a backwash is not possible. Therefore, the feed water must be free from particles. In case of fouling or scaling a clean in place (CIP)

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can be carried out with acid and caustic, chlorine is not possible. The pre-treatment membranes protect the RO from organic and /or colloid fouling, retain particles and bigger molecules. The retained impurities can be removed easier by backwash and chemical cleaning compared to RO.

Table 38. CS9: Technical details of all tested membranes; TMP: transmembrane pressure.

Parameter	Unit	Conventional UF	Ultra-tight UF	Open NF	RO
Name		UFC-LE (Pentair)	Not commercial (Pentair)	HFW1000 (Pentair)	LCHR-4040 (Dupont)
MWCO	kDa	150	4	1	
Pore-size	nm	5-20		1-10	
Material		PVP and PES	modified PES and PES	modified PES and PES	
Membrane area	m²	40	40	40	8.7
Design flux	L/(m² h)	60 – 120	20 – 60	15 – 30	22.3
Design recovery	%	65-85 (crossflow & 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

Due to the much smaller molecular weight cut-off of the ultra-tight UF or open NF compared to a conventional UF, colloids and large organic molecules such as biopolymers can be better retained. The assumption was, that then the risk of fouling of RO is reduced and also the frequency of CIP procedures, the replacement of membranes and the demand for chemicals.

One part of the tests was the impact of coagulant. Batch tests and dosing before UF/NF were carried out.

The impact of UF / ultra-tight UF/ open NF on RO operation was investigated including a cross-evaluation of potential savings regarding chemical cleanings and exchange of membranes, obtaining higher flux and recovery rates as well as less flushing cycles, etc. on the RO side against additional operational efforts and costs on the UF/NF side.

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The TRL of the operation of the membrane plant increased from 6 to 7. The TRL of the u-t UF increased from 5 to 7. The capacity of the technology can be easily adapted and expanded to the needs of the customer. The technology can directly be used for the treatment of the mWWTP of Kalundborg and can also be transferred to other cities and countries. Here the legal basics have to be checked in advance.

Technical requirements for the implementation and operating conditions

Table 39 shows the feed water requirements for the application of UF/NF membranes. If the chemical composition of the WWTP effluent does not comply with the indicated ranges, a pre-treatment prior to the inflow to the unit is recommended, e.g. sand filtration or dual media filtration.

Table 39. CS9: Typical feed water conditions to apply UF/NF membranes [Pentair 2022].

Parameter	Units	Feed water requirements
Temperature	°C	0 – 40
pH	-	2 – 11
Particle size	µm	< 300
Total suspended solids	mg/L	< 200
Ozone	mg/L	0
Free chlorine	mg/L	< 500

Sand filter/ dual media filter

Granular media filtration as pre-treatment before UF/NF is normally not used. Only in case a filter is built for other reasons, such as the protection of the Baltic Sea or the implementation of the revised urban wastewater directive, its implementation can make sense.

In this pilot study a continuously operated sand filter (SF) and a dual media filter (DMF) were tested upstream of the treatment train. The filter removed the organic particles most likely originating from the biotech industry and improved the continuous operation of the pilots by protection against particle peaks.

The dual media filter was preferred in this case because manual cleaning and easy operation were the advantages of this type of filter.

Pre-filter before UF/NF

To prevent the pilot units from clogging by small particles, a pre-filter unit (for the pilots: Amiad®) with a 300 µm screen and automatic flushing (every 4-5 min) was installed upstream of each UF/NF. This is also absolutely necessary for the full-size plant.

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UF/NF

During pilot operation different operational settings were tested. These settings are listed in Table 40.

Table 40. CS9: Tested operational settings.

Parameter	Unit	Conventional UF		Ultra-tight UF	Open NF
		crossflow [1)]	dead end		
Flux	L/(m² h)	30 – 40 – 50	50 – 60 – 65	20 – 25 – 30 – 32.5 – 35	20 – 22.5 – 25 – 30 – 32.5
Recovery	%	65 – 75 – 85	100	75 – 80	50 – 75 – 80
Crossflow velocity	m/s	0 – 0.15 – 0.25 – 0.3	0.4	0.3 – 0.4 – 0.5	0.3 – 0.4

1) With discharge of concentrate

Additional different frequency of backwash (BW) and automatic chemical cleaning (CEC) were used and as acid HCl and citric acid were tested. Dosing of coagulant was tested as batch test and before UF/NF with different amounts of chemical. A long-term test with coagulant should be carried out to check if the cleanable of membranes can be improved with this dosing.

2.7.2. Results of new approaches

Results of the feasibility study and expected technology performance (KPIs)

The removal efficiencies of the tested UF, u-tUF and NF membranes are shown in Figure 61 for selected parameters such as the turbidity, chemical oxygen demand (COD), total organic carbon (TOC), sulphate, total hardness and conductivity.

The turbidity retention ranged for all three membranes between 90% and 95%. The COD retention, the TOC retention and the sulphate retention were higher for the NF membrane with 73%, 50%, and 25%, respectively compared the UF and u-t UF membranes with roughly 25%, 15% and 5% and below. The removal capacity for the total hardness and conductivity were only slightly different with 15% and 5% for the NF and 5% and 3% for the UF and u-t UF, respectively.

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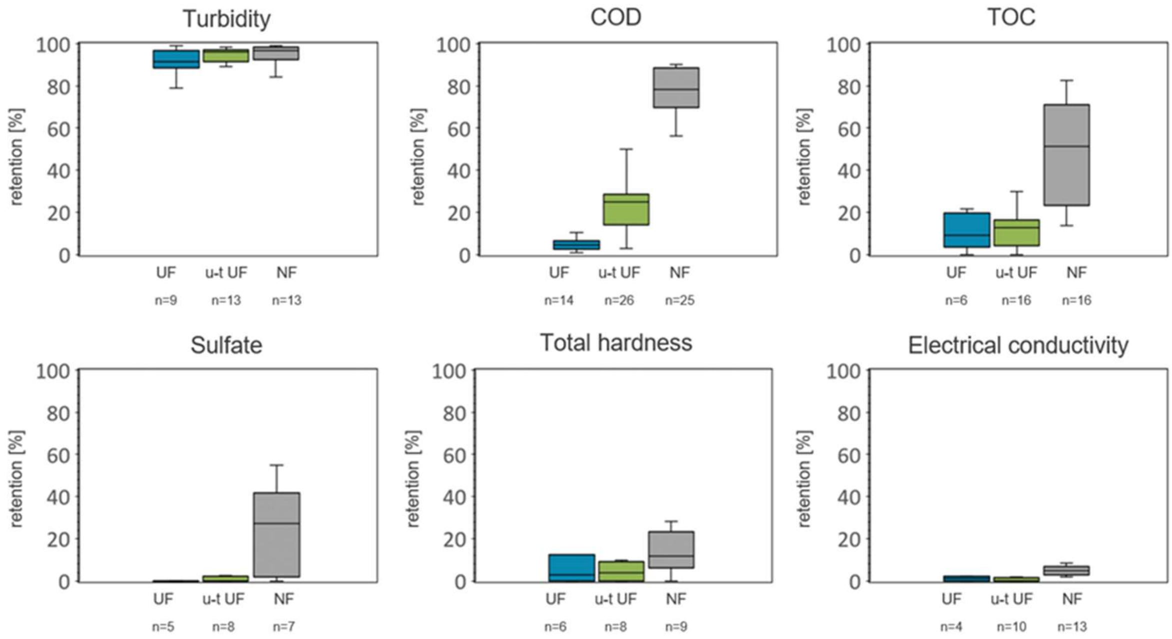

Figure 61. CS9: Performance of UF, u-t UF and NF: removal of selected parameters with number of samples.

The removal of turbidity was high for all membranes as expected. The NF retained the organic compounds better than the UF and the u-t UF, what was also expected, because the lower molecule weight cut off (MWCO) of the NF (1 kDa) compared to the u-t UF (4 kDa) and UF (150 kDa) leads to a better retention. Surprisingly, even though the MWCO of the u-t-UF was smaller than that of the UF, the retention of the TOC was in a similar range for both. One reason therefore might be a very small size of the organic compounds enabling them to pass through pores sizes with a MWCO of 4 kDa. Ezugbe et al., 2020 and Poirier et al. 2023 indicate a higher retention of COD, however due to the industrial wastewater fraction coming from biotech and pharmaceutical industry, it is likely that very small organic compounds contained in the wastewater mix. This was also observed by Alturki et al. (2012). Hence, the small molecular weight enables them to pass through the u-t UF membrane unit. In conclusion, the NF performed much better than the UF and u-t UF. The advantage of the u-t UF of a lower MWCO compared to the UF, however, was very small and showed only negligible or slightly better removal rates.

Comparison of operational parameters and performance of UF, u-t UF and NF

In the Table 41 the possible and preferred operation parameters are shown for all tested membranes.

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Table 41. CS9: Comparison of operational parameters: UF, u-t UF and NF; bold: preferred settings; green: positive results, yellow: medium results, red: negative results.

Parameter	Unit	Conventional UF		Ultra-tight UF	Open NF
		crossflow 1)	dead end		
MWOC	kDa	150		4	1
Flux	L/(m^2 h)	**40**	50 – **60** – 65	15 – 37	15 – 32.5
Recovery	%	65 – **85***	**95 – 99****	75 – **80**	50 – **75*** – **80****
TMP 2)	Bar	0.03 – 0.4	0.04 – 0.9	0.15 – 1.0	1.0 – 2.7
Energy consumption	kWh/m^3	0.09	0.10	0.12	0.18
Retention TOC	%	11		12	49
Retention COD	%	5		23	78
Frequency of CIP RO (with biocide)	1/a	---	3 – 5	---	0 – 4

* without SF

** with SF

1) With discharge of concentrate

2) For preferred settings; depending on feed water quality and time after CEC

The conventional UF reached a flux of 40 L/(m^2 h) and a recovery efficiency of 65-85 % in crossflow mode and a flux of 60 L/(m^2 h) and a recovery efficiency of 95-99% in dead end mode. The u-t UF and the open NF reached max. 37 L/(m^2 h) and max. 32.5 L/(m^2 h) as flux and 80 % recovery each. The measured transmembrane pressures (TMP) ranged for the UF, u-t UF and NF between 0.03 – 0.9 bar, 0.15 – 1 bar and 1 – 2.7 bar, respectively. The TMP depends on the feed water quality and operational time after a chemical cleaning (CEC) and correlates with the energy consumption. The CIP frequencies of the RO were calculated using the performance parameter permeability of RO. For the calculation, only the period with biocide treatment was used. Using the UF as pre-treatment, the cleaning demand of the RO would be 3-5 times per year and with NF, it would be 0-4 times per year.

Hence, the highest flux and recovery efficiencies were reached with the conventional UF. For the design of a full-scale system, the high fluxes and recovery rates allow for lower membrane areas and hence, for a lower number of modules to reach the same amount of reclaimed water. Also, the lower concentrate production by the UF should be noted.

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 advantage. The calculated frequency of the CIPs was only marginally higher for

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the RO with the UF as pre-treatment, not being a major disadvantage. For the membranes with a lower MWCO (NF and u-t UF) the retention of organic compounds is higher than for the UF, this is also shown in Table 41.

All tested membranes were suitable as pre-treatment of the RO treating the clarified mixture of biotech and municipal wastewater. However, the most suitable pre-treatment for a full-scale system seems to be the conventional UF, because of its lower energy demand and its high flux as well as recovery efficiency.

Due to particle peaks the quality of feed water differs from time to time. The operational parameter of the units can be adapted, e.g. with lower flux and recovery for this time period. Also, the cleaning frequency can be increased. With this the UF showed to be easier to maintain and the impurities could be easier removed than for the u-t UF and NF.

Coagulant dosing and with different dosing rates in batch tests and upstream of the UF were also tested (0.5–3 mg/L Fe). However, this did neither increase the flux nor enhance the retention of any dissolved compounds. The possible benefit of coagulation on long-term fouling should be tested further. The exchange of HCl as cleaning agent to citric acid led to a more continuous operation.

Although the new u-t UF will not be selected for a full-size plant, the KPIs were determined and are shown in Table 42.

Table 42. CS9: Comparison of key performance indicators for the novel ultra-tight UF membrane.

Parameter	Unit	Estimated Value	Measured value	
Turbidity Permeate	NTU	< 0.2 1)	< 0.1 ± 0.05	n=10
Total suspended solids Permeate	mg/L	< 0.2 1)	0.7 ± 0.6	n= 7
Bacteria (*E. coli*)	log removal	> 4 – 6 1)	4 ± 0.003	n=3
Intervals backwash of u-t UF	min	30 – 60 2)	60	
Intervals of CEC of u-t UF	h	24 2)	24	
Intervals of CIP of u-t UF	months	3 – 12 2)	0	
Intervals CIP of subsequent RO	weeks	1 – 2 2)	1 (without biocide treatment)	

CEB – chemical enhanced backwash

CEC – chemical enhanced cleaning

CIP – cleaning in place

1) Estimated based on X-Flow XF64 Membrane (Pentair 2022)

2) Jährig et al. 2023

The new u-t UF decreased the turbidity as expected to < 0.2 mg/L. The content of total suspended solids could not be measured with the required accuracy, the limit of

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quantification was 0.5 mg/L and the fluctuations in the measurement were even higher (± 0.6 mg/L). A log removal of 4 for *E. coli* was reached, this is within the expected value.

The backwash interval was 60 min and the interval for CEC was daily as expected. However, CIPs were not necessary for the u-t UF. The interval of the subsequent RO cleaning was weekly without biocide. With a regular biocide treatment, the interval is expected to be much longer.

Performance of reverse osmosis

In Figure 62, the permeability of the RO is shown as one indicator for the RO performance. A decrease of permeability means a lower performance of RO.

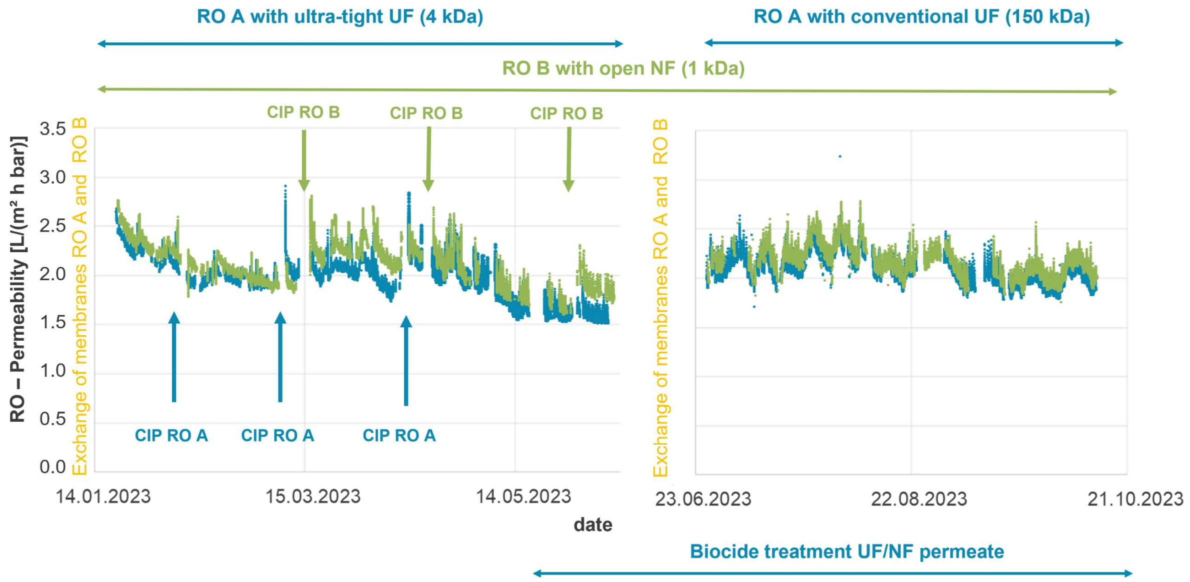

Figure 62. CS9: Permeability of RO with different pre-treatment membranes: (left): u-t UF and NF, (right): UF and NF with an intermittent biocide treatment of the RO feed (tanks and pipes).

In the first period, the addition of biocide was avoided to test, if a denser pre-treatment membrane might replace the normally required biocide treatment (Figure 62, left graph). A clear decrease in permeability was visible for both membrane combinations, u-t UF with RO and NF with RO. To increase the permeability, the clean-in-place CIP was used. However, after each CIP only a small increase in permeability was observed. In the second period (Figure 62, right graph), biocide was regularly dosed to the feed tank and pipes to the RO. This way, the permeability remained on a stable level for both membrane combinations, UF with RO and NF with RO. The clear decreases in permeability in the first period suggest, that fouling processes took place on the RO membranes of both combinations. Because the CIPs removed only a part of the impurities, irreversible fouling occurred. However, in the second period no permeability decreases were observed and hence, no fouling occurred as a very likely

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result of the biocide dosing. Thus, the fouling in the first period resulted mainly from microbial activity.

Dosing of biocide is the most commonly used technique to avoid fouling processes on and in the RO, but it is also very hazardous (Da-Silva-Correa et al. 2022). Denser membranes with a MWCO of 4 kDa and 1 kDa as pre-treatment of the RO feed alone could not avoid biofouling under the conditions in this study. However, the biocide had a major positive effect on the performance of the ROs, while the different pre-treatment membranes showed no high difference. Furthermore, due to the biocide dosing, no CIPs were necessary for four months. This means, as long as biocide is dosed, also a membrane such as the conventional UF membrane is suitable to maintain a stable operation of a RO membrane.

To prevent the RO before scaling an antiscalant dosing is absolutely necessary. During pilot operation the antiscalant Kemira KEMGUARD was used with a dosing rate of 7.4 mg/L. Chemical cleaning (Cleaning in place, CIP) of the RO was carried out if required. Here, the recommendations of the supplier were confirmed.

Selection of best pretreatment option for reverse osmosis

All tested membranes were able to provide a suitable feed quality for the RO. Even though the NF showed a higher retention in terms of organic compounds compared to the u-t UF and NF, the ability to prevent fouling processes on the subsequent RO was very similar for all membranes. Hereby, it was much more important to dose biocide than to use a certain density of the pre-treatment membrane. Hence, the newly developed u-t UF had no clear advantages over the conventional UF and the open NF. Furthermore, the conventional UF showed a high robustness against the fluctuating feed quality and easier handling. Due to particle peaks the quality differs from time to time. The operational parameter of the units can be adapted, e.g. with lower flux and recovery for this time period. Also, the cleaning frequency can be increased. With this the UF showed to be easier to maintain and the impurities could be easier removed than for the u-t UF and NF.

For an up-scaled system, the conventional UF requires fewer modules and produces less concentrate, which has to be returned to the WWTP. Also, Poirier et al. 2023 concluded that UF-RO is the best combination.

Upscaling of the selected treatment train

For the design of a full-scale plant the software WAVE was used. In Figure 63, the schemes of the possible 2-stage RO or 3-stage RO are displayed. It is assumed that several lines with 100 L/h permeate production will be operated. The membrane area of one module is 40 m² of FILMTEC™ BW30 PRO – 400 (DuPont). With a design flux of 22.3 L/(m² h) (tested in pilot plant) and the design temperature of 21°C about 20 pressure vessels with 6 membranes each (120 in total) per line are necessary. For the 2-stage RO a recovery of 65% can be reached and an energy consumption of 0.78

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kWh/m³ was calculated. For a 3-stage RO a higher recovery of 68% can be reached with also a higher energy consumption of 0.83 kWh/m³.

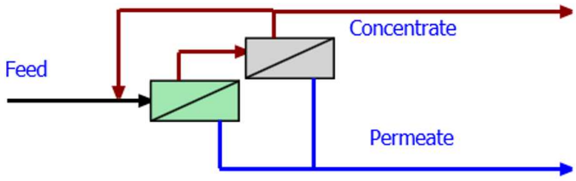

Figure 63. CS9: Design of full-size RO, left: 2-stage RO; right: 3-stage RO.

During the tests with biocide treatment before RO once per week 4 mg/L DBNPA solution were dosed. This was a sufficient concentration for a very good performance of the upstream RO. He et al. 2016 found a dosing of 20 mg/L for 60 min/d to optimise the operation and reduce frequency of CIP of RO. The RO manual (Dupont 2023) recommended an intermittent dosing of 10 – 30 mg/L for 0.5 – 3 h every 5 days. To prevent scaling on the RO membrane, antiscalant must be dosed before RO cartridge filter. During pilot time 7.4 mg/L Kemira KEMGUARD was dosed. For a full-size plant about 3 – 5 mg/L is assumed.

The RO elements should be cleaned when one or more of the parameters are applicable (Dupont 2023):

i. The normalized permeate flow drops 10%
ii. The normalized salt passage increases 5 – 10%
iii. The normalized hydraulic pressure difference increases 10 – 15%

The cleaning frequency was calculated with the hydraulic pressure difference and is shown in Figure 64.

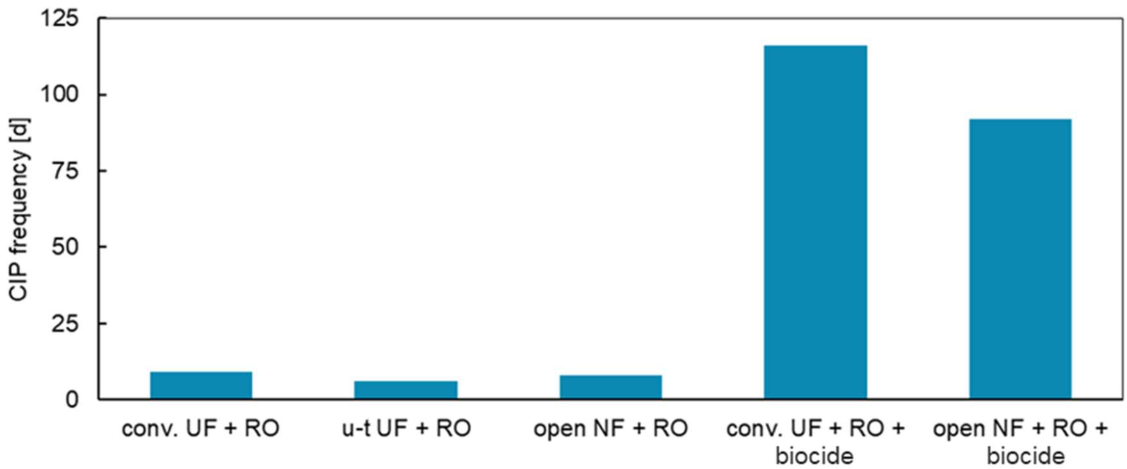

Figure 64. CS9: CIP frequency of RO calculated using the hydraulic pressure difference.

The operation without biocide shows a very short operation time of RO until the next CIP for all tested pre-treatment membranes. A CIP is necessary about weekly. With

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biocide the frequency of CIP could be increased until 3 – 5/a with UF and 0 – 4/a with NF. A clear benefit of biocide dosing is visible.

In Table 43 an overview about the possible treatment of the waste streams is shown. The possibilities were compared using Life Cycle Assessment (LCA). This can be found in D2.2.

Table 43. CS9: Possible concentrate and brine treatment.

Concentrate UF	Brine RO	
Back to inlet WWTP • With dead end operation very small amount of wastewater	Discharge into Green Belt • High flow rate • Higher concentrations but same load + antiscalant • Authorities might decrease discharge thresholds	Further treatment (e.g. zero liquid discharge) • High tech • High energy consumption

Cooling water quality

The results of the water quality of the RO permeate in comparison to the required water quality for cooling purposes and the current used lake water are shown in Table 44.

Table 44. CS9: Quality of RO permeate and comparison with cooling water quality and currently used lake water quality; green: cooling water quality reached/lake water quality exceeded, yellow: cooling water quality can be reached with common post treatment.

Parameter	Unit	Goal: cooling water 1)	RO 2)	n	Raw lake water 4)
Aluminium	µg/L	< 500	< 44 3)	38	n.a.
Calcium	mg/L	> 20 / < 500	< 0.5	23	110
Carb. hardness	°dH	< 4 / < 20	< 0.1	16	n.a.
Chloride	mg/L	< 50 / < 250	13.4	25	n.a.
COD	mg/L	< 5	< 5	30	26
Copper	µg/L	< 500	< 0.5	25	n.a.
Conductivity	µS/cm	50 – < 3000	5.8	29	620
Iron	mg/L	< 0.1 / < 0.5	< 0.05	19	0.06
Magnesium	mg/L	< 100	0.1	24	n.a.
pH		7 – 9	6.4	28	8.2

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Parameter	Unit	Goal: cooling water 1)	RO 2)	n	Raw lake water 4)
Sulphate	mg/L	< 50 / < 600	1.0	24	75
TDS	g/L	< 1.8	0.06	21	n.a.
Total hardness	°dH	0.1 – < 8	< 0.1	23	17.4
TSS	mg/L	< 5	0.6	18	1.6
Turbidity	FNU	< 1	< 1 3)	48	1.5
E. Coli	MPN/100 mL	Absence	< 1	19	7
Legionella	CFU/L	< 100	< 10	11	n.a.

1) Royal Decree 1620/2007, 2007; VDI 2047 Bl. 2, 2015 & VDI 3803 Blatt 1, 2019; Niewersch et al, 2016.

2) Independent from pre-treatment, results of both ROs together

3) Already reached after UF/NF

4) single measurement

The results refer to the combined permeates of both ROs, independent from their pre-treatment method. The requirements for the water quality were defined by using technical guidelines (VDI 2047 Bl. 2, 2015 & VDI 3803 Blatt 1, 2019; Niewersch et al, 2016) and the Spanish Regulation for Water Reuse (Royal Decree 1620/2007), because specifically for Denmark neither a regulation nor guidelines were available. For aluminum, the carbonate hardness, chloride, copper, the conductivity, iron, magnesium, sulphate, total dissolved solids (TDS), the total hardness, total suspended solids (TSS) and the abundance of *Escherichia coli* as well as *Legionella*, the RO permeate complied with the required water quality. However, for calcium and the pH, the concentrations were even below the required concentration ranges. The water quality parameters of the RO permeate were lower for all parameters compared to the raw lake water.

Regarding calcium and the pH, higher concentrations are required. This can be easily achieved using a common post-treatment process such as chemical stabilization. Fulfilling all requirements of the technical guidelines and the Spanish regulation, shows that all treatment trains are suitable to produce the required water quality. Niewersch et al, 2016; Van Houtte, Verbauwhede, 2012 + 2013; Nahrstedt 2020 made similar observations, which also used an UF and RO as well as a NF and RO to successfully produce fit for purpose water for industrial use (e.g. cooling). The requirements for the abundance of *Escherichia coli* as well as *Legionella* were already reached in the permeate of the UF and NF (< 1 FNU/ < 10 CFU/L), what is also typical for both membrane types and has been frequently observed in practice (Bodzek et al., 2019). Because no specific Danish guidelines or regulations exist, the RO permeate water quality was compared to that of the currently used cooling water, which is raw lake water. Also, in this regard, all treatment trains provided even a better water quality than

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that which is currently used. Hence, the produced fit for purpose water is very suitable to be reused as cooling water in industry.

Comparison of baseline situation with ULTIMATE solution

The results of the pilot plant showed that the proposed treatment train, consisting of a dual media filter, ultrafiltration and reverse osmosis, was the best option to treat the WWTP effluent in an energy efficient manner to achieve cooling water quality. The water quality achieved with the proposed treatment train was even better than the quality of the raw lake water, as already shown in the chapter "Cooling water quality". The implementation of a full-scale water reclamation plant is planned with the aim of completely replacing the lake water used for cooling purposes. The planned production of fit-for-purpose water from the mWWTP effluent is expected to be around 3.5 million m^3/a or even more.

2.7.3. Conclusions

Lessons learned from technology operation and symbiotic relationship (technical risks & measures)

The mWWTP effluent is highly influenced by the treated industrial wastewater coming from the biotech industry. Due to its chemical composition, the risk of fouling processes taking place on membranes is quite high. Therefore, despite the availability of mature membrane technologies for water reclamation, we conducted tests on different pre-treatment options for the feed water to the RO to reduce fouling processes. Our pilot study yielded valuable insights that can contribute to future decisions:

The operation of a membrane plant in general is possible with all tested membranes. More effort should be put into proper pre-treatment. Here, a continuously operating DynaSand filter and a dual media filter were tested. Both showed good protection against changing feed water quality. Also, the pre-filter of UF/NF was absolutely necessary for the operation.

The operational parameters of the units can be adapted according to the feed water quality, e. g. a lower flux and recovery for the time period treating effluent of poor quality. Also, the cleaning frequency can be increased. The system requires more intensive maintenance.

It is recommended to test the coagulant dosing upstream of the UF. The possible benefit of coagulation can be the easier removal of impurities on membranes during backwash and CEC. The use of citric acid instead of HCl as cleaning agent led to a more continuous operation.

The production of "fit-for-purpose" water is possible. All chemical and micro-biological parameters for cooling water can be easily met with UF-RO treatment. The produced quality is better than the current used lake water. The lake water can be easily substituted. Microorganisms like *E. coli* and *Legionella are* effectively retained

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by UF/NF. A common posttreatment of RO permeate is necessary for pH adjustment and stabilization.

For the reduction of fouling on the RO membrane the benefit of denser membranes as pre-treatment is very small. The greatest influence was the biocide treatment of tanks and pipes before RO. For a full-size plant a biocide dosing upstream of RO should be foreseen. Antiscalant upstream of RO is essential to prevent the scaling of RO due to high hardness of water.

The additional costs due to denser membrane are not justified. The denser the membrane, the more energy is required. Also, due to lower flux and recovery, more membrane area is necessary. This leads to higher investment costs.

The main challenge of implementing this scheme is to obtain the official permission of the authorities, since Denmark opted out of the Water Reuse Regulation and the new Danish Water Law does not allow for water reuse. Thresholds for water quality such as for cooling water still need to be defined in the Danish legislation.

Risks and measures

Before installation of such a fill-size plant the authorisation to use the water must be obtained. Also, the concept for the treatment or discharge of the brine must be clarified. For this, all stakeholders must be involved into planning. The distribution of the fit-for-purpose water additional requires an extra pipeline.

Best practices and recommendations for technology design and operation in the symbiotic frame

- There are only very minor differences between the tested pre-treatment membranes.
- No clear advantages of the newly developed u-t UF membrane were found.
- The conventional UF is robust against the fluctuating feed quality, easier in handling, consumes less energy, fewer modules are necessary and the UF produces less wastewater. These are clear advantages of the conventional UF.
- A pre-filter before UF with automatic flush is absolutely essential.
- A sand filter or dual media filter before the membrane plant is recommended. This leads to an equalization of the fluctuating inlet qualities (particles), it is easier to operate the UF and improve the water for discharge into the Great Belt.
- The discharge or treatment of the brine of RO has to be discussed and clarified with the authorities before installation.
- There must be legal certainty for the use of treated wastewater.

Advantages

- Technology already in operation in full size in Europe
- Size can be adapted easily according to water demand

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- Produced water quality is very high

Disadvantages

- Energy consuming technology
- High investment costs
- Brine discharge or treatment has to be clarified in advance
- Extra pipeline for distribution required
- In Denmark: no existing or planned regulations for water reuse

Crucial factors for technology implementation and its optimal performance

- For water reclamation from municipal WWTP effluent containing a high fraction of treated biotech wastewater, a suitable treatment train is a sand filter or dual media filter combined with a conventional ultrafiltration and a reverse osmosis.
- Cooling water quality can be reached with this treatment train after a chemical stabilization of the RO permeate.
- Biocide treatment is necessary to avoid organic fouling processes on the RO.
- There must be legal certainty for the use of treated wastewater.
- There must be a concept for the treatment or discharge of the brine.
- Because the costs of the fit-for-purpose water are high, it should be only implemented when no alternative water resources are available (see also D2.2).

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3. Summary and conclusion of water recycling concepts

3.1. Summary and comparison of similar technologies

ULTIMATE is focused on water smart industrial symbioses (WSIS) between the industrial sector and services providers of the water sector. The WSIS approach is the basis for a successful implementation of circular economy technologies, because one partner produces the resource for the circular economy solution and the other partner has the demand for the recovered product. Thus, they cooperate for their mutual benefits.

In seven of nine ULTIMATE case studies, water-related technologies were conceptualised, developed and demonstrated. These technologies have demonstrated the multiple uses of municipal and industrial wastewater from the agro-food processing, beverage, heavy chemical/petrochemical, and biotech industries, obtaining fit-for-purpose water for industrial (mainly, cooling water) and agricultural uses (irrigation).

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Table 45. Summary of water-related technologies with TRL increase in ULTIMATE, flowrates, KPIs and their feasibility.

CS	Technology	TRL	Water type produced or saved	Inflow flowrate/ capacity	Water recovery rates (KPIs)	Feasibility and/or successful operation
1	UF+RO+MD (nZLD)	5→7	Cooling water	0.5-1 m³/h (UF) 22-49 m³/h (RO) 375-545 L/h (MD)	>20% for UF+2-pass RO, depending on UF recovery (KPI: >20%)	Successful pilot operation for UF and RO MD permeate does not fulfil with water quality requirements
1	Ammonium adsorption on zeolite	5→6	Cooling water	150-250 L/h	6% energy consumption reduction (KPI: 10%)	Successful pilot operation.
2	Electrodialysis/Capacitive electrodialysis	6→7	Irrigation water	1-4 m³/day	16% reduction of the primary freshwater input to the greenhouse consortium for irrigation purposes (KPI:20%)	Successful pilot operation. KPI not achieved but 60-90% recovery of water from the greenhouse wastewater
3	SWMM+RO	5→8	Irrigation water or industrial water, depending on the quality	450-800 L/h	>35 % (KPI: 35%)	Successful pilot operation
4	VAC adsorption/extraction+ coagulation+AOP reactors+Small bioreactor platform	6→7	Irrigation water	10 m³/day	90% recovery of water (compared to the water present in industrial by-product) (KPI: 90%)	Successful pilot operation
5	NF+RO	7→9	Cooling water	0.8-1.6 m³/h	>15% (KPI: 10%)	Successful pilot operation
7	RO	5→7	Cleaning water and cooling water	0.1 L/s	>66% (KPI: 40%)	Successful pilot operation
9	Dynasand/media filter + UF/u-t UF/NF+RO	5→7	Industrial water	0.6-4.8 m³/h (UF/NF) 0.2 m³/h (RO)	66% reduction of freshwater consumption through reuse of the treated wastewater. (KPI: 40%)	Successful pilot operation

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For all technologies, that started at an TRL of 5-7, the demonstration at case study level was successful and increased their TRL to 7, 8 and 9.

Table 45 shows the comparison of the expected water KPI achievements at the beginning of ULTIMATE compared to the actual achieved water savings at case study levels.

Table 46 presents the benefits and challenges of the water recovery technologies. For some of the technologies, there are more benefits than challenges. In addition, most of the challenges often refer to water pre-treatment required, concentrate streams management and high operational and investment costs. The successful lab and pilot demonstrations in ULTIMATE were important steps to evidence the high potential and feasibility of the technologies. Not all the technologies are still mature enough to be marketed directly, but they are very well suited for further investigations and more demonstration projects to accelerate their market-uptake.

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Table 46. Benefits and challenges of the ULTIMATE water recovery technologies.

CS	Technology	Benefits	Challenges
1	UF+RO+MD (nZLD)	• Reclaimed water can be produced with UF+2-pass RO • UF and RO are mature technologies, available on the market (easier to scale-up)	• Membrane technologies have high energy consumption • MD is not a mature technology, not available on market at industrial scale • Concentrate effluents must fulfil with discharge limits and it can be a high constraint in case of scaling up the proposed technologies (post-treatment required)
1	Adsorption on zeolite	• Lower energy consumption in comparison with RO • Easy operation and maintenance	• Low ammonium adsorption capacity • Regeneration solution (waste) management • High footprint in case of scaling up at industrial scale • This technology does not removed conductivity
2	Electrodialysis/Capacitive electrodialysis	• Both technologies can meet stringent water quality requirements, essential for safe and effective irrigation • Lower energy consumption than RO	• Current costs of ion-exchange membranes and energy can be a bottleneck for uptake as compared to other technologies • Discharge management (concentrate) is still a (technical & regulatory) issue • Complete solution (incl. disinfection) still to be assessed (dealing with food products) • Discrepancy in water demand and supply – buffering or storage (e.g. subsurface) required, legislation for such storage unclear

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CS	Technology	Benefits	Challenges
			• Risk-averse nature of farmers/end-users, trust and incentives needed
3	SWMM+RO	• SWMM models developed were able to predict with relatively high resolution both the quantity and quality of the seawater infiltrated into the sewage networks of the municipalities • Modelling+digital tools have the potential to increase reused water in the region for both industrial and agricultural use • Reverse osmosis enables to produce high-quality water	• For developing accurate hydraulic models that aim to detect peaks of saline intrusion, it is of highly importance to acquire robust local data with high resolution (high number of sensors required) • Correct distribution of sensor for wastewater flow and conductivity it is necessary to develop modelling and digital tools • Reverse osmosis has a high energy consumption • RO concentrate management
4	VAC adsorption/extraction+ coagulation+AOP reactors+small bioreactor platform	• Polyphenols can be recovered • Technologies in portable container	• High cost of the global treatment • Adsorption material high-priced • Adsorption material and regeneration solutions (waste) management • Annular reactor achieves partial mineralization • SBP expensive technology
5	NF+RO	• NF protects RO from organic and /or colloid fouling, due to the much smaller molecular weight cut-off of the open NF membranes compared to a conventional UF • Reduction in the number of clean in place (CIP) or membrane replacements of RO • Reduction of the operating costs	• NF is less competitive in CAPEX and OPEX than conventional UF • Pre- or post-treatment can be required, depending on the water characteristics • RO concentrate management required

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CS	Technology	Benefits	Challenges
		• NF is a valid technology for meeting water reuse regulatory requirements, but for salinity removal a further RO step is needed	
7	RO	• Mature technology • It can be integrated with nutrients recovery processes	• Inlet stream must be solids free (pre-treatment required) • High energy consuming technology • RO concentrate management required
9	Dynasand/media filter + UF/u-t UF/NF+RO	• Technology already in operation in full size in Europe • Size can be adapted easily according to water demand • Produced water quality is very high	• Energy consuming technology • High investment costs • Brine discharge or treatment has to be clarified in advance • Extra pipeline for distribution required • In Denmark: no existing or planned regulations for water reuse

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3.2. Recommendations for best practice for the implementation and application of the technologies under different process conditions

ULTIMATE is focused on water smart industrial symbioses (WSIS) between the industrial sector and services providers of the water sector. The WSIS approach is the basis for a successful implementation of circular economy technologies, because one partner produces the resource for the circular economy solution and the other partner has the demand for the recovered product. Thus, they cooperate for their mutual benefits.

The results included in this report highlight their potential for their scaling-up and exploitation in several cases of study, and, furthermore, their replicability. However, when scaling a solution or proposing its replication, it is essential to consider not only the technical aspects but also a range of other key factors, including economic, regulatory, political, and social considerations.

Lessons learned and replication potential

Most of the lessons learned outlined in this document focus on the more technical and scientific aspects of the proposed technologies in the various case studies, although economic and regulatory aspects are also discussed.

Initially, a comprehensive characterization of the starting wastewater stream and the desired final quality to be achieved is required. It may be advisable to first evaluate the proposed technologies at a laboratory scale to define an optimal operating range, and based on these conditions, move to a pilot scale.

The pilot-scale technologies that have been evaluated were tested in an industrial environment, using real water. This has sometimes involved treating a water stream with variability not observed at the laboratory scale, leading to operational challenges in the different processes, and even, concluding that a pre-treatment or post-treatment would be necessary in case of scaling-up the solution.

Finally, industrial wastewater technologies combined with digital tools, as in CS3, enables more efficient, sustainable, and controlled water treatment processes. This approach allows companies to reduce water usage, meet environmental standards, and maximize operational efficiency. Integrating industrial wastewater treatment with digital tools promotes a more sustainable and optimized water management strategy.

Policy relevance of the results

The results of several cases of study have shown that policy recommendations and changes are crucial for fostering water reuse and promote a water circular economy model.

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Clarifying responsibilities and developing consistent guidelines for water reuse licensing and service provision across the EU are essential to ensure effective practices. Increasing treated wastewater quality requirements under the revised can simplify water reclamation processes, potentially eliminating the need for additional advanced treatments. Future regulations should establish minimum standards for non-agricultural uses, enhance risk assessment, and promote research on innovative water reuse technologies and practices.

Economic considerations

The economic aspects, both CAPEX and OPEX, are critical when scaling up an industrial water treatment train, but they have only been estimated in a few case studies. In some instances, the significant difference in treatment flow rate between the pilot plant and a future industrial-scale facility must be considered. Therefore, for cases with the highest potential, it may be advantageous to conduct tests using a demonstration plant with a larger flow rate treatment capacity, allowing for more accurate estimates of investment and operational costs.

Business models, funding, and pricing are critical for successful implementation of some of the assess solutions in ULTIMATE, with multi-user systems enhancing technical and economic viability.

Public engagement and awareness

The strategic agenda proposes comprehensive coverage of all water reuse types, emphasizing safety, environmental impact assessment, and the integration of reclaimed water into local water balances based on regional circumstances. However, public engagement and awareness are essential to overcoming negative perceptions and increasing acceptance of reused water.

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Annex 1: CS2 experimental conditions to determine the performance of the ED set-up

Overview of the tested conditions for the ED set-up (above) and (C)ED pilot (below).

No.	Membrane (10 cell pairs)	Experiment (Stopped after 4 h or at dilute EC< 0.2 mS/cm)	Voltage (V)	Flow rate (cm/s)	Volume (Diluale:Concentrate)
1	Fujifilm Type 10	Limiting current density	0-10 (LCD recorded at 8V)	1	01:01
2	Fujifilm Type 10	Baseline	6	1	01:01
3	Fujifilm Type 10	Higher concentrate	6	1	05:01
4	Fujifilm Type 10	Decrease retention time	6	2	01:01

Experiment (One pass)	Feed	Feed EC (mS/cm)	Diluate EC target (mS/cm)	Operating parameters
Control	NaCl synthetic feed	9	1, 0.5, 0.2, <0.2	For standardization, voltage variable, water recovery 50 %
(C)ED	De Vlot wastewater	1,9 1, 0.5, 0.2, <0.2		Crossflow velocity 5.12 cm/s, stack staging 2-stages, voltage 6-16 V, water recovery 60 %, 80 % 90 %

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ULTIMATE

WATER SMART INDUSTRIAL SYMBIOSIS

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