

Accompanying document to
Deliverable 1.2
Operational demo cases

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Technical References

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¹ PU = Public

PP = Restricted to other programme participants (including the Commission Services)

RE = Restricted to a group specified by the consortium (including the Commission Services)

CO = Confidential, only for members of the consortium (including the Commission Services)





Document history

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Executive Summary

The European industry is the [largest water consuming sector after agriculture](#), with a significantly larger water footprint than residential/urban areas. They also face a fierce competition worldwide, with limited domestic resources. Moving to a circular economy (CE) paradigm that valorises a wide range of water-embedded resources: water, energy, nutrients and high added-value compounds, will future-proof European industries, climate-proof European society and safeguard the environment. Water Smart Industrial Symbiosis (WSIS) as a particular form of CE applicable to industrial contexts promises a new potential by systematically looking to reuse wastes between industries as raw materials. WSIS promises benefits from lower costs as well as new types of revenues, exploiting “waste” management not only as a legal obligation but as a new business opportunity.

WSIS is a novel approach with as of yet limited applications. In ULTIMATE, WSIS between the industrial sector and service providers of the water sector are demonstrated at significant scales thus creating an evidence-based approach for successful WSIS implementation anchored on real-world cases.

Therefore, at nine case studies distributed across Europe and Israel, the ULTIMATE consortium has established nine WSISs. They develop and demonstrate 24 pilot plants, which recover water, materials and/or energy.

Deliverable D1.2 is a demonstrator type deliverable and shall show, that the ULTIMATE pilot plants are operational. Therefore, presentations showing the operational pilot plants will be accessible on the ULTIMATE webpage at the case study section (<https://ultimatewater.eu/demonstration-cases/>). This document accompanies the presentations which are meant to be the main evidence for D1.2 and shows the progress until M37.

Prior to the pilot plant implementation, eight WSISs conducted laboratory experiments. In total, 17 different laboratory experiments and/or investigations of already existing facilities are accomplished to better understand the circumstances of the real environment and to learn more about the type of technology before it is up-scaled from laboratory to pilot scale. Eleven of the 17 investigations are already completed and four are close to be completed (75-90%).

Until M24, five pilot plants or (parts of) treatment trains were operational. Three of them are related to water recovery at the case studies in Nafplio (CS4), Lleida (CS5) and Kalundborg (CS9), one of them to material recovery in Lleida (CS5) and the last one to energy recovery in Karmiel (CS6).

Until M27, five additional plants were operational. Two of them refer to water recovery in Lleida (CS5) and Tain (CS7), one to material reuse in Rosignano (CS3) and two to energy recovery and reuse in Shafdan (CS6) and Tain (CS7).





Until M30, six additional pilots were put into operation. Two relate to water recovery and are located in Tarragona (CS1). One was implemented in Nieuw Prinsenland (CS2) and refers to water recovery and material recovery. The other three pilots focus on material recovery and were put into operation in Napfljo (CS4), Tain (CS7) and at the Chemical Platform of Roussillon (CS8).

Since M33, four additional pilots are operational in Lleida (CS5) and in Karmiel (CS6) referring to energy recovery and material recovery, respectively.

In the course of the project, three pilots had been included in the description of work in addition. Due to their later inclusion, they have a different time planning and hence, two of them are not operational yet. They will be located in Rosignano (CS3) and are expected to be operational in September 2023 and in January 2024. Furthermore, the full-scale electrostimulated anaerobic reactor (ELSAR, CS5) has also another time planning and is expected to be operational in September 2023 as the industrial pilot plant of CS8 is at the Chemical Platform of Roussillon.

Until all pilot plants will be operational, a very close monitoring of the case studies will be done by the WP1 management team with the case study leaders and the risk officer via regularly meetings. In addition, the presentations referring to D1.2 will be further updated every three months until every pilot plant will be operational. Those results will then be reported in the best practice guidelines (D1.3, D1.4 and D1.5).

D1.2 is the basis for the demonstration of the ULTIMATE solutions and for the generation of valuable data. Those data will be needed for the technology evidence base (D1.7), for the best practice guidelines (D1.3, D1.4, and D1.5) and also for the assessments of our circular economy solutions (D2.2 and D2.5). Those results will contribute to find suitable strategies for the replication of our concepts and thus, be the basis for the overall exploitation strategy (D5.9).

Hence, the EU-added value of this deliverable is its contribution to crucial deliverables that will foster and boost circular economy solutions in the European industry and the water sector. The collection and open access presentation of the technologies in the technology evidence base (D1.7) will support decision makers and investors to gain a fast overview of the opportunities and proven concepts of circular economy. Together with the Marketplace (D5.5), the technology evidence base can significantly contribute to the transition from a linear to a circular economy in Europe.

ULTIMATE promotes circular economy solutions that are in line with the ambitions of the European Green Deal (European Commission 2019) its Action Plan for Circular Economy (European Commission 2020) to reduce strongly our greenhouse gas emissions, to provide clean water, to maintain healthy soil, make industry resilient and produce cleaner energy. This deliverable (D1.2) presents technologies that can be applied in the frame of the Regulation (EU) 2020/741 on minimum requirements for water reuse, the Regulation (EU) 2019/1009 laying down rules on the making available on the market of EU fertilising products and the Directive (EU) 2018/2001 on the promotion of the use of energy from renewable sources.





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Table of Contents

1.	INTRODUCTION	9
2.	OPERATIONAL DEMO CASES	13
2.1.	CS1: TARRAGONA	15
2.2.	CS2: NIEUW PRINSENLAND	25
2.3.	CS3: ROSIGNANO	40
2.4.	CS4: NAFPLIO	59
2.5.	CS5: LLEIDA	80
2.6.	CS6: KARMIEL & SHAFDAN	121
2.7.	CS7: TAIN	152
2.8.	CS8: CHEMICAL PLATFORM OF ROUSSILLON	169
2.9.	CS9: KALUNDBORG	185
3.	SUMMARY AND CONCLUSION	199
4.	LITERATURE REFERENCES	202





Abbreviations

AAT	Advanced anaerobic treatment (immobilised high-rate anaerobic reactor)
AnMBR	Anaerobic membrane bioreactor
AnBTMBR	Anaerobic biofilm treatment membrane bioreactor
AOP	Advanced oxidation process
ATES	Aquifer thermal energy storage
BES	Bioelectrochemical systems
CE	Circular economy
COD	Chemical oxygen demand
CS	Case study
CTG	Cross-cutting technology group
ELSAR	Electrostimulated anaerobic reactor
GAC	Granulated activated carbon
HTC	Hydrothermal carbonisation
MBR	Membrane bioreactor
NF	Nanofiltration
nZLD	Near zero liquid discharge
PE	Population equivalent
RO	Reverse osmosis
SBP	Small bioreactor platform
SCWE	Supercritical water extraction
SME	Small and medium enterprises
TEB	Technology evidence base
UF	Ultrafiltration
WSIS	Water smart industrial symbiosis
WWTP	Wastewater treatment plant

Table of tables

Table 1	Overview about the ULTIMATE solutions: relevant for D1.2.....	10
Table 2	Overview about the resources and pilot plants referring to each case study	13
Table 3	Overview about the progress regarding the pilot plants.....	200





1. Introduction

In ULTIMATE, water smart industrial symbioses (WSIS) between the industrial sector and service providers of the water sector are established to implement and operate innovative circular economy solutions. The WSIS are considered to be the basis for a successful implementation of those 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. At nine case studies distributed across Europe and Israel, the ULTIMATE consortium develops and demonstrates 24 pilot, full-scale and industrial-scale plants, which recover water, materials and/or energy (Table 1).

Hereby, eight, six and nine pilot plants refer to water recovery, energy recovery and material recovery and reuse, respectively. One additional pilot refers to both water and material recovery. The grey coloured technologies refer either to only concept studies or to early warning systems, data-driven matchmaking platforms and/or control systems. Those systems need more time than only 24 months to be investigated and developed. Therefore, they have been excluded from D1.2 what was already indicated in the grant agreement. Their results will be part of the deliverables D1.3, D1.4 and D1.5 *New approaches and best practices for closing the water, material and energy cycles*.

Deliverable D1.2 is a demonstrator type deliverable and is supposed to show, that the ULTIMATE pilot plants are operational. To document that this status has been achieved, for every case study a presentation containing pictures and/or videos of the operational pilot plant is accessible on the ULTIMATE webpage in the case study section (<https://ultimatewater.eu/demonstration-cases/>). Currently, 20 pilots are operational and four pilot plants are still under construction. For those, the presentations will be updated every three months until all pilot plants are operational. This document accompanies these presentations that are the main evidence for D1.2 and shows the progress until M37.

The baseline conditions of each case study were described in detail in D1.1 (Kleyböcker et al. 2021a) showing the opportunities and the demands for the implementation of the circular economy ULTIMATE concepts. In D1.8 (Kleyböcker et al. 2022), the concepts are explained in detail and discussed in the context of similar research projects and concepts. Hence, D1.2 is the next step towards the overall goal of ULTIMATE to show the successful implementation of the concepts and to derive best practise guidelines for closing the water, material and energy cycles at the case studies within the symbioses clusters. Those results are and will be presented in detail in D1.6 & D1.7 *Technology Evidence Base* (D1.6, Kleyböcker et al. 2021b) as well as in the deliverables D1.3, D1.4 and D1.5 *New approaches and best practices for closing the water, material and energy cycles*.





Table 1 Overview about the ULTIMATE solutions: relevant for D1.2 are the blue (water recovery and reuse), green (material recovery and reuse) and yellow (energy recovery and reuse) coloured technologies

CS Name		Water Smart Industrial Symbiosis						Explanation of colour code/scale indication				
		Industrial Sectors			Service Providers							
		AgroFood	Beverage	Chemical/Petrochemical	BioTech	Municipal utility	Multi-industry utility	Specialist SME	providing water services	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												
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 adsorbent for wastewater treatment TRL 4 → 7	
3	Rosignano (IT)								Advanced oxidation process pilot plant with advanced monitoring system using locally produced peroxides TRL 4 → 6		Use of industrial byproducts in a clarification-flocculation plant TRL 4 → 6	
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 combining 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	





CS Name		Water Smart Industrial Symbiosis			Explanation of colour code/scale indication		
		Industrial Sectors		Service Providers	WATER RECLAMATION AND REUSE	NUTRIENT & MATERIAL RECOVERY & REUSE	ENERGY & HEAT RECOVERY & REUSE
		AgroFood	Beverage	Chemical/Petrochemical			
		BioTech	Municipal utility	Multi-industry utility			
				Specialist SME providing water services	NO PILOT PLANT --> NOT PART OF D1.2	COMBINATION OF THE CS4 PILOT PLANTS FOR WATER & MATERIAL	
Technologies applied & Circular Economy contributions							
5	Lleida (ES)				Water reuse after treatment with AnMBR and ELSAR with fit-for-purpose post-treatment: NF & RO: TRL 7 → 9; AOP & UV: TRL 7 → 9; <i>Online Monitoring: TRL 5 → 7</i>	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 and utilisation for treatment steps 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 (<i>Integration of solutions of other sites with TRL > 6</i>)	Data driven control system to increase energy efficiency through a synergetic operation of an industrial and municipal WWTP TRL 5 → 8





The results will also be used for the different assessments and analyses in WP2, they will be used as a basis for potential replication ambitions (WP5), for the identification of policy gaps for the implementation of such technologies (WP4) and the marketability of their products (WP5).

Hence, this deliverable contributes to crucial deliverables that will foster and boost circular economy solutions in the European industry and the water sector. The collection and open access presentation of the technologies in the technology evidence base (D1.7) will support decision makers and investors to gain a fast overview of the opportunities and proven concepts of circular economy. Together with the Marketplace (D5.5), the technology evidence base can significantly contribute to the transition from a linear to a circular economy in Europe.

ULTIMATE promotes circular economy solutions that are in line with the ambitions of the European Green Deal (European Commission 2019) its Action Plan for Circular Economy (European Commission 2020) to reduce strongly our greenhouse gas emissions, to provide clean water, to maintain healthy soil, make industry resilient and produce cleaner energy. This deliverable (D1.2) presents technologies that can be applied in the frame of the Regulation (EU) 2020/741 on minimum requirements for water reuse, the Regulation (EU) 2019/1009 laying down rules on the making available on the market of EU fertilising products and the Directive (EU) 2018/2001 on the promotion of the use of energy from renewable sources.





2. Operational demo cases

In ULTIMATE, 24 pilot plants are developed and will be demonstrated at nine case studies to showcase innovative circular economy solutions (Table 2). Furthermore, CS1 - CS8 conducted laboratory experiments, before they implemented their pilot plants. In total, 17 different laboratory experiments and/or investigations of already existing facilities were and still are accomplished to better understand the circumstances of the real environment and to learn more about the type of technology before the implementation and in parallel to the operation pilot-scale and full-scale plants.

Table 2 Overview about the resources and pilot plants referring to each case study

Case study	Resources	Treatment trains
CS1 Tarragona (ES)	Municipal wastewater and industrial wastewater from the petrochemical complex	Water recovery: Reverse osmosis and membrane distillation Water recovery: Ammonia removal via zeolites
CS2 Nieuw Prinsenland (NL)	Drain water from greenhouses; residual and geothermal heat	Water recovery and material recovery: Reclamation of greenhouse drain water using electrodialysis and recovery of nutrients including test beddings and demo greenhouse
CS3 Rosignano (IT)	Byproducts from industry for reuse in water treatment	Material recovery and reuse: Pilot scale adsorption system & use of byproducts Material recovery and reuse: Advanced oxidation process pilot plant using locally produced peroxides with an advanced monitoring system Material recovery and reuse: Use of industrial byproducts in a clari-flocculation plant
CS4 Nafplio (EL)	Wastewater from fruit processing industry	Water recovery: Filtration, advanced oxidation and small bioreactor platform Material recovery: Plant to recover antioxidants
CS5 Lleida (ES)	Wastewater from brewery & municipal wastewater	Water recovery: Nanofiltration & reverse osmosis as part of the post-treatment Material recovery: Solar-driven hydrothermal carbonisation demo plant Water recovery: Advanced oxidation & UV light treatment Energy recovery: Anaerobic membrane bioreactor Energy recovery: Solid oxide fuel cell Energy recovery: Pilot-scale and full-scale electro-stimulated anaerobic reactor (ELSAR)
CS6 Karmiel/Shafdan (IL)	Wastewater from olive oil production, slaughterhouses and wineries & municipal wastewater	Energy recovery: Biogas production from olive mill wastewater: high rate anaerobic reactor Energy recovery: High rate anaerobic reactor with membrane filtration incl. AC Material recovery: Plant to recover polyphenols





Case study	Resources	Treatment trains
CS7 Tain (UK)	Wastewater from whiskey distillery	Water recovery : Reverse osmosis to treat AnMBR effluent Energy recovery : Heat recovery unit Material recovery : Struvite and ammonia sulphate recovery units
CS8 Chem. Platform Roussillion (FR)	Wastewater from chemical industry	Material recovery : Laboratory scale and full-scale sulphur recovery unit
CS9 Kalundborg (DK)	Wastewater from pharma & biotech industry and municipal wastewater	Water recovery : Treatment train for water recovery involving a novel ultrafiltration membrane or open nanofiltration unit.

In the following chapters, the progress per case study referring to the relevant subtasks for D1.2 are shown in detail.





2.1. CS1: Tarragona

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
1	1.2.1	RO + MD; ammonia removal via zeolites	100%	100%	100%	Nov 22



D1.2 Operational demo cases

CS1 - Tarragona

EURECAT & AITASA





CS1: Tarragona

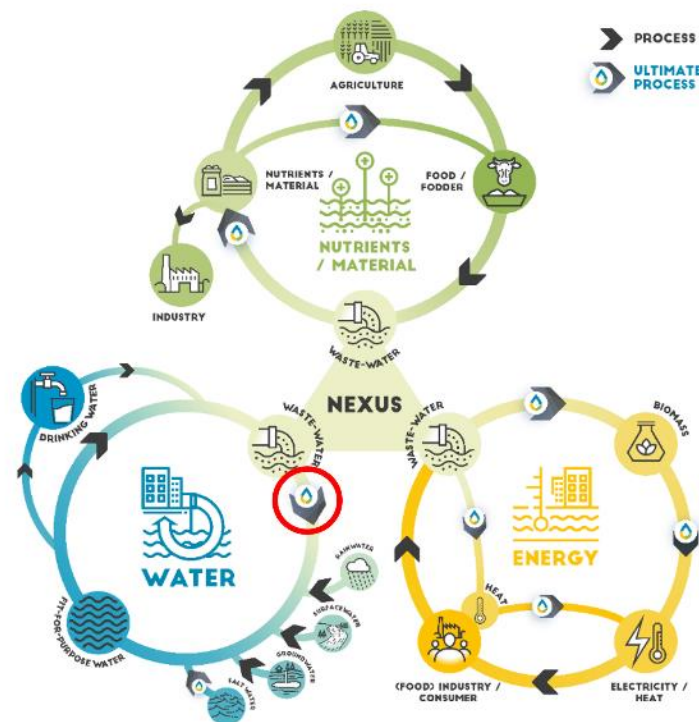
Lead partner:



Other partners:



2



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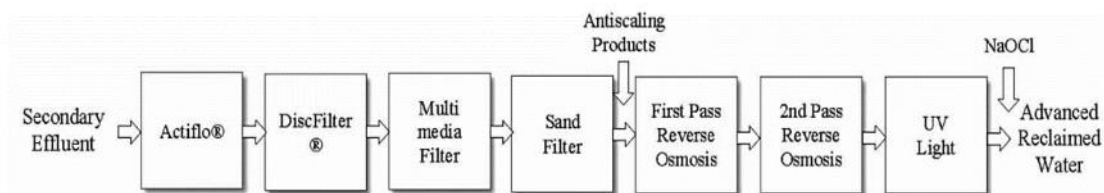


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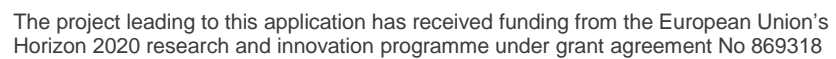
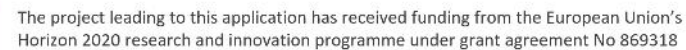
CS1: Situation before Ultimate

- ✓ The Tarragona and Vilaseca-Salou wastewater treatment plants (WWTPs) were interconnected by a 4-km pipeline to ensure that the AWRP can be supplied with enough secondary effluent from either or both WWTPs. Secondary effluent undergoes a basic **water reclamation process** at the **WRP** (1021 m³/h average inlet flow rate), consisting of a ballasted clarification step (Actiflo® unit), followed by disc filtration (DiscFilter® unit), multimedia filtration and sand filtration. The effluent undergoes an advanced reclamation process including a two-pass reverse osmosis (RO) treatment processes and disinfection, using ultra-violet light and chlorine, before it enters the reclaimed water distribution system. Furthermore, chemical reagents such as coagulant, flocculent and antiscaling are added to enhance the plant performance.



- ✓ On the other hand, in order to meet future water requirements (BREF limits), an **industrial wastewater treatment plant (iWWTP)** has been commissioned in April 2022 to polish the aggregated wastewater from the petrochemical complex and to produce reclaimed water for the complex (1348 m³/h average water flow rate). The technology train to be implemented in these new facilities will be:
- Dissolved air flotation (DAF)
 - Biological membranes (MBR)
 - Granular activated carbon (GAC)







CS1: Objectives of the Ultimate solutions

OBJECTIVE:

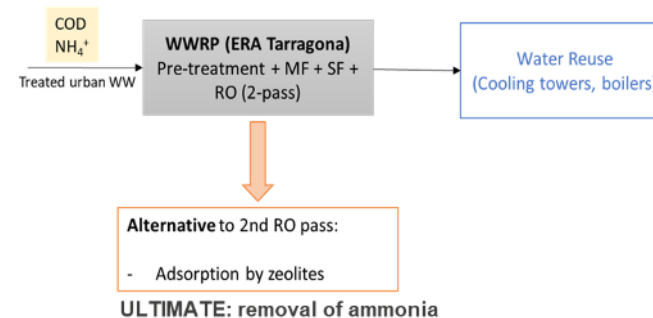
Increase reclaimed water availability for the complex by 20%:

→ Current WWRP:

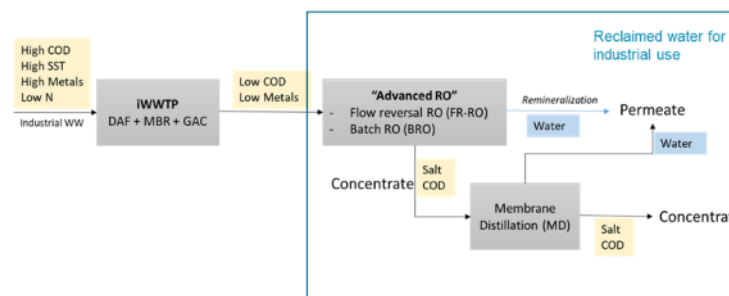
- Increase water recovery of the current WWRP with nZLD technologies
- Remove the ammonium with low-cost technology (zeolite adsorption)

→ Future iWWTP:

- Defining a novel tertiary treatment with nZLD technologies (reverse osmosis and membrane distillation) to obtain reclaimed water



ULTIMATE: Proposed WWRP scheme to maximize water recovery (near ZLD)



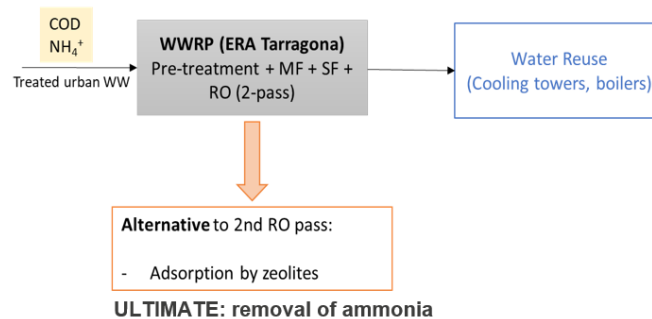


CS1: Status/progress

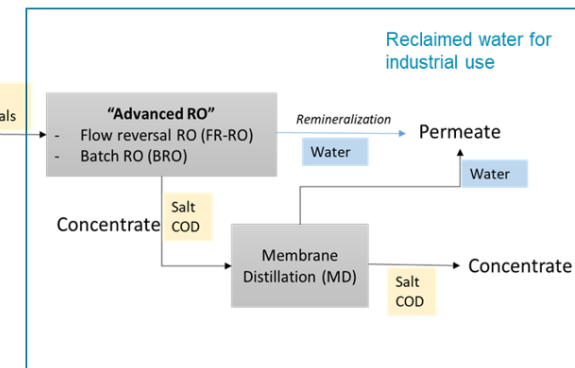
Subtask: 1.2.1 Increasing reclaimed water availability of the petrochemical complex of Tarragona (ES)

Baseline technology: WWRP (pre-treatment+MF+SF+2-pass RO), iWWTP in operation since April 2022.

Ultimate solution to foster circular economy:



ULTIMATE: Proposed WWRP scheme to maximize water recovery (near ZLD)



TRL: 5→7 (membranes), 5→6 (adsorption on zeolites)

Capacity: 12 m³/day

Quantifiable target: <20% reduction of fresh water through reuse of treated wastewater; 10 % reduction of energy demand

Status/progress:

- Bench scale experiments finished (UF, RO, MD and adsorption on zeolites)
- Pilot plants operational: UF+RO+MD & Zeolites



CS1: Pictures/videos of the new technologies

Subtask: 1.2.1 Increasing reclaimed water availability of the petrochemical complex of Tarragona (ES)

Optimal operation conditions obtained experimentally at bench scale → Pilot plant design

UF bench scale experimental set-up



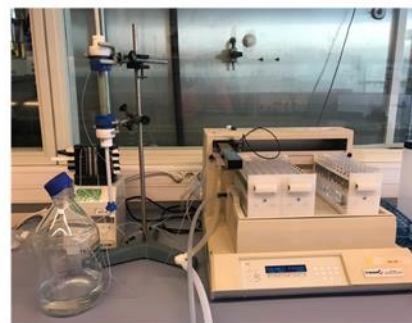
RO bench scale experimental set-up



MD bench scale experimental set-up



Zeolite adsorption bench scale experimental set-up



7



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CS1: Operational procedures and methodologies

Subtask: 1.2.1 Increasing reclaimed water availability of the petrochemical complex of Tarragona (ES)

- Work at bench scale is finished.
- Pilot plant:
 - All equipment from supplier 1 (UF+RO+zeolites) and supplier 2 (MD) delivered and installed in AITASA facilities.
 - Hydraulic tests with UF + RO units with clean water → OK
 - Adsorption with zeolites equipment needs some modifications → RO permeate storage tank (feed) and valves for sampling points.
 - Start-up with real water scheduled for 21st November week and training for all the technologies.



Maritim container and tanks



UF unit (left) and RO unit (right)



Adsorption with zeolites column



Operation monitoring

8



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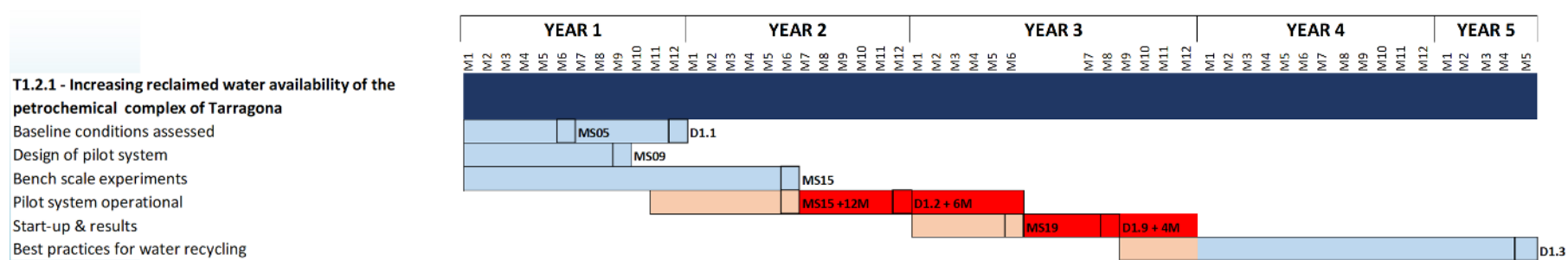


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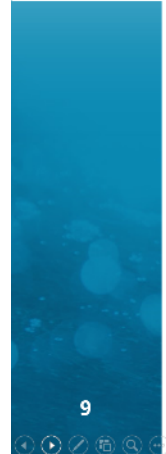


CS1: Task 1.2.1 – Timeline

Subtask: 1.2.1 Increasing reclaimed water availability of the petrochemical complex of Tarragona (ES)



→ Pilot system is operational since November 2022 (M30)



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Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity



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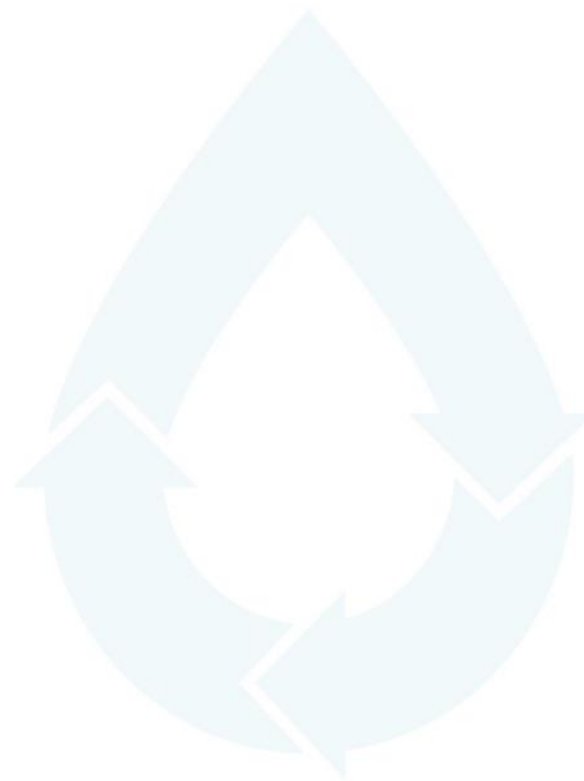
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2.2. CS2: Nieuw Prinsenland

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
2	1.2.2	Reclamation of water using electrodialysis	75%	100%	100%	Nov 22
	1.3.1	Feasibility study: HT-ATES	No pilot plant --> excluded from D1.2			
	1.4.1	Recovery of nutrients: test beddings & demo greenhouse	75%	100%	100%	Nov 22



D1.2 Operational demo cases CS2 – Nieuw Prinsenland

KWR





CS2: Nieuw Prinsenland

Lead partner:

KWR

Collaborators:

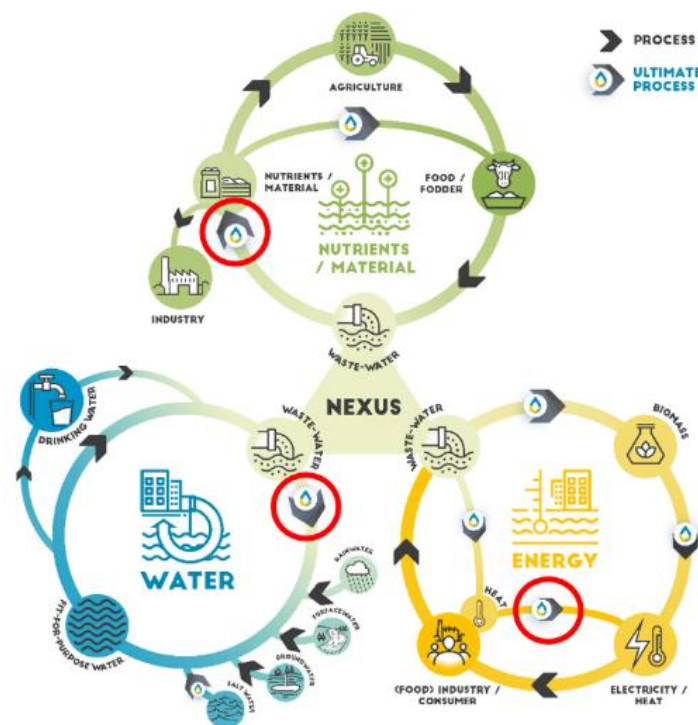
Coöperatieve Tuinbouw Water Zuivering
De Vlot



2



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CS2: Situation before Ultimate

Situation at the Coöperatieve Tuinbouw Water Zuivering de Vlot (November 2020)

The current status is that the wastewater treatment plant is operational. From January 2021, they can remove crop protection agents from the wastewater. The cooperative aims to start working towards reusing/recovering water and nutrients from 2021 onwards.

Process steps at the site:

Prefiltration by vibrating and rotating filters: suspended solids removal

Coagulation in sedimentation buffers: P removal

Sand filtration with glycerol dosage: N removal
(not operational as high nutrient load results in clogging and hence too high maintenance)

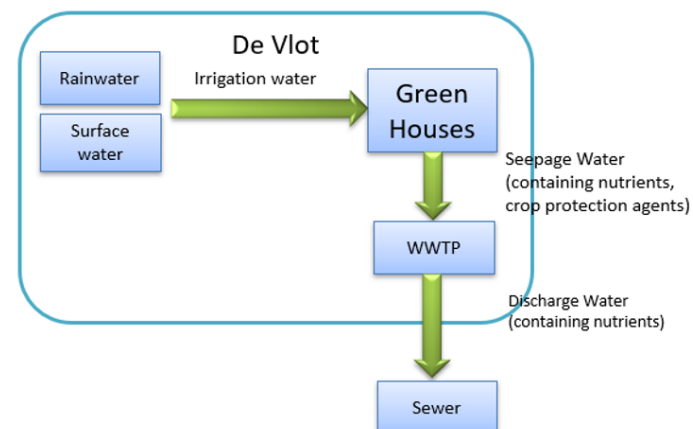
Activated carbon: crop protection agent removal

3



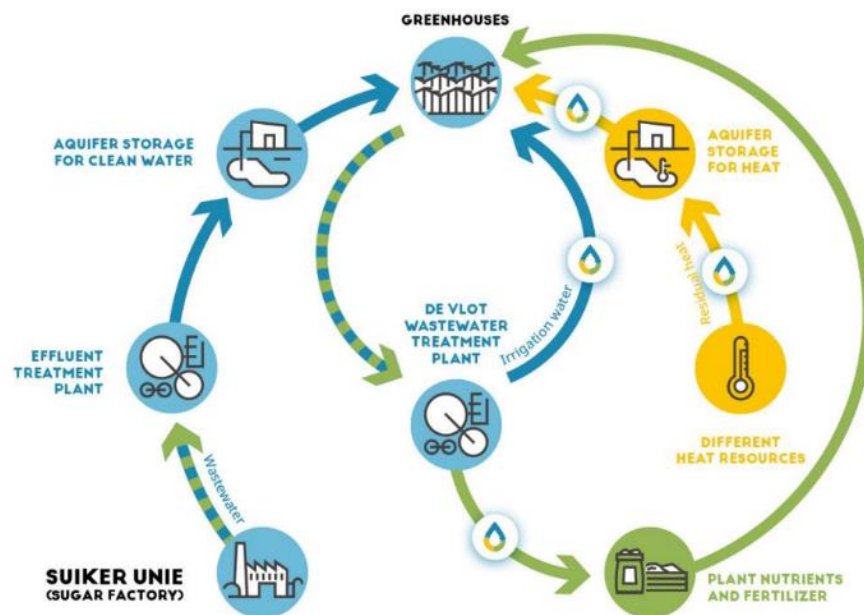
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Current hydrological cycle De Vlot





CS2: Objectives of the Ultimate solutions



4



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CS2: Objectives of the Ultimate solutions

WATER: Task 1.2.2 (KWR) Optimizing water reclamation from agro-food industries in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

The main aim of this task is to facilitate the reuse of wastewater from greenhouses with a view on optimizing the water reclamation. To do so, an extensive analysis of the treated wastewater will be conducted. Then, an adequate treatment will be determined supported by a quantitative microbial risk assessment (WP2), so that water suitable for irrigation purposes (main objectives - free of pathogens, low in sodium) can be supplied for irrigation in the greenhouses.

In order to validate a reliable way of removing plant diseases from the water, the reuse of this water will be investigated on pilot scale in a demo-greenhouse.

Finally, a full-scale treatment solution will be designed based on the previous results and the ones of the economic analysis (WP2).

5



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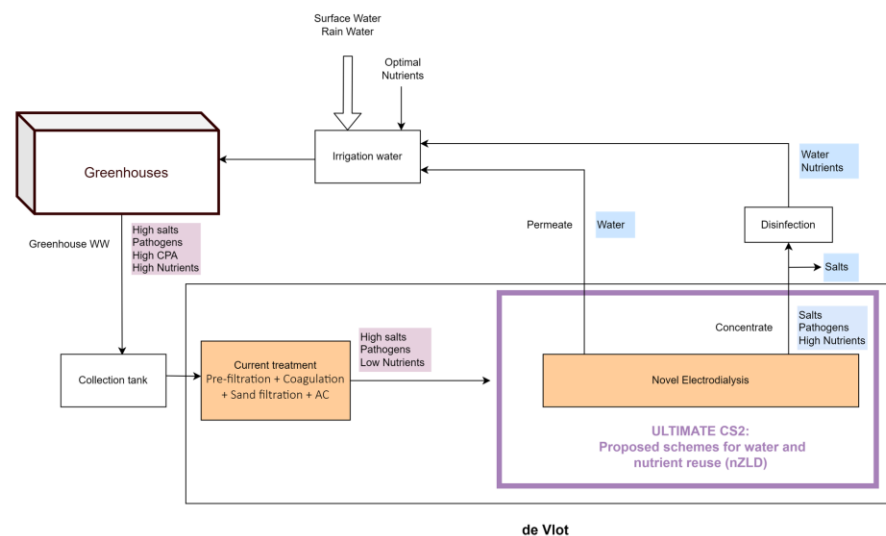
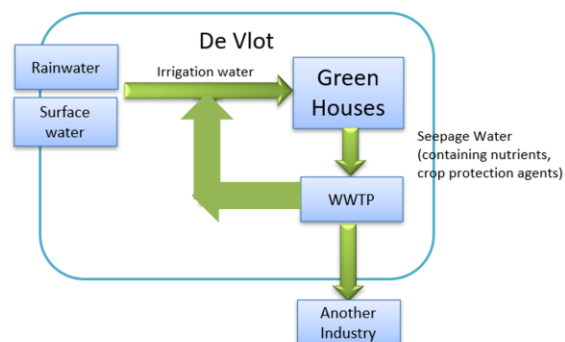


CS2: Subtask 1.2.2 Status/progress

Subtask: 1.2.2 Optimising water reclamation from agro-food industries in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

Baseline technology: no water reuse so far

**Ultimate solution to foster circular economy:
New hydrological cycle**



TRL: 4 → 6

Capacity of the pilot plant: 0.1 m³/day

Quantifiable target: ambition beyond the project: 25% reduction of freshwater through water reuse (700 m³/d)

Status/progress:

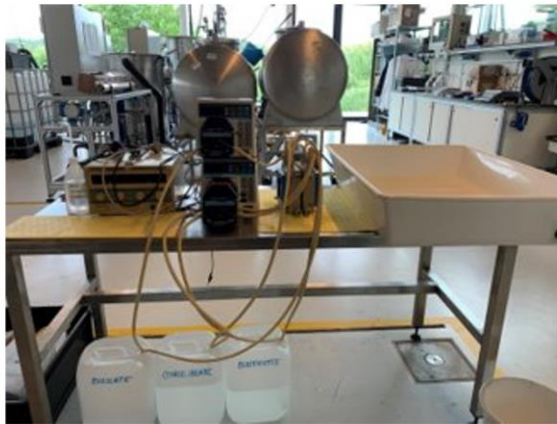
- Performance validation for Proof of Concept on laboratory scale being finalized.
- Detailed pilot design – completed
- Construction of pilot plant – completed and pilot plant - operational



CS2: Pictures/videos of the new technologies

Subtask: 1.2.2 Optimising water reclamation from agro-food industries in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

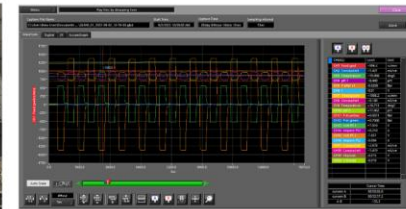
Electrodialysis experiments



Lab experiments ongoing at KWR



Pilot experiments ongoing at Ghent University



Lab experiments to continue for remainder of project to support pilot.



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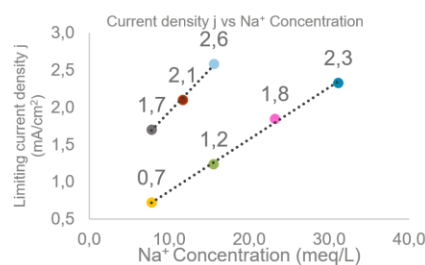


CS2: Results of the laboratory experiments

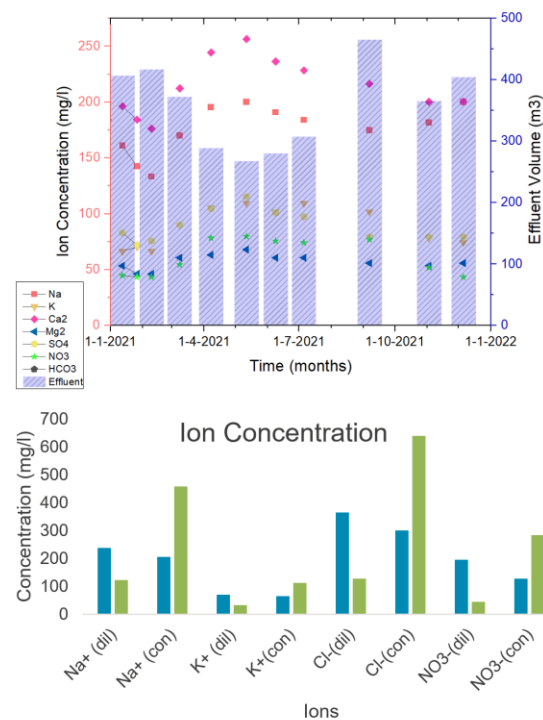
Subtask: 1.2.2 Optimising water reclamation from agro-food industries in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

Work to date has focused on validation of the methodology on lab scale and confirming the required performance in selective removal of sodium can be achieved.

- 60% reduction in EC (1 ms/cm) (~50% Na removal)
- On-going optimization lab experiments with Greenhouse simulated and real wastewater



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CS2: Subtask 1.2.2 – Timeline

Subtask: 1.2.2 Optimising water reclamation from agro-food industries in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

T1.2.2 - Optimising water reclamation from agro-food industries in N. Prinsenland

Baseline conditions assessed

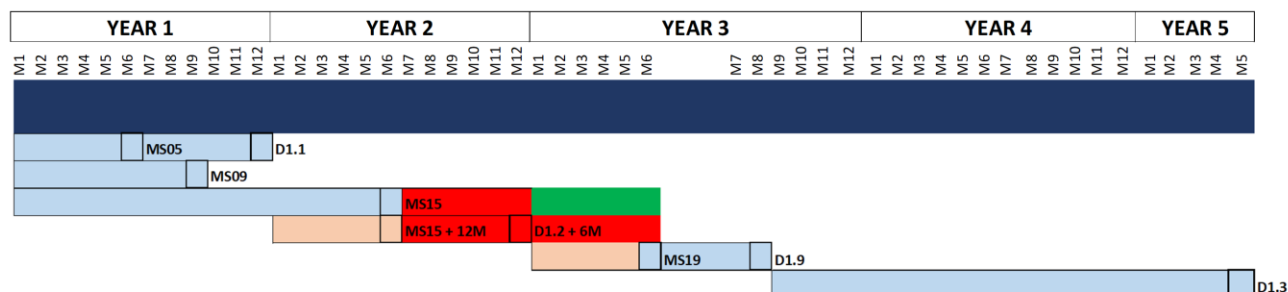
Design of pilot system

Bench scale experiments

Pilot system operational

Start-up & results

Best practices for water recycling



- Pilot system operational since November 2022 (M30)
- First results from pilot plant operation obtained
- Still enough time to complete the pilot experiments

Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity





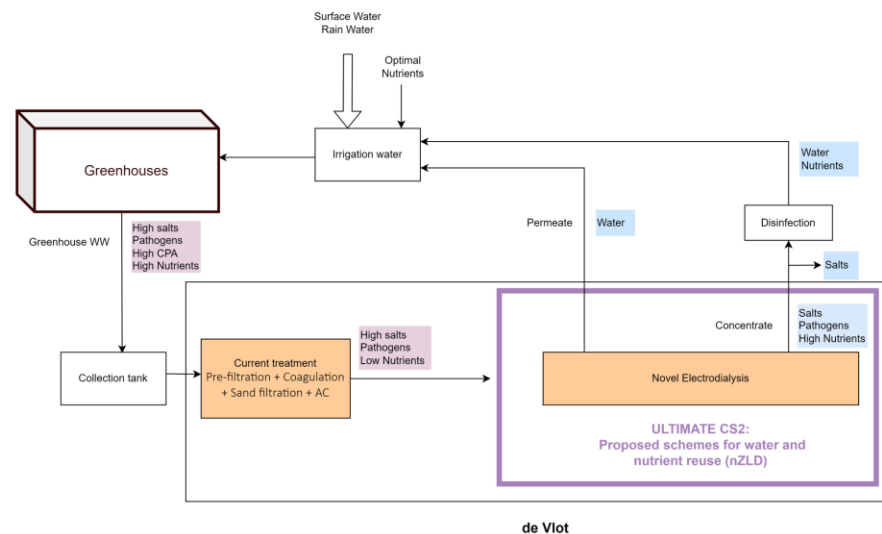
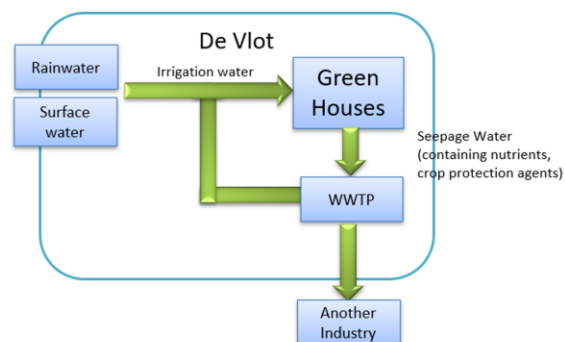
CS2: Subtask 1.4.1 Status/progress

Subtask: 1.4.1 Recovery of nutrients from greenhouse wastewater in N. Prinsenland and Coöperatieve Tuinbouw Water
Zuivering de Vlot

Baseline technology:

Ultimate solution to foster circular economy:

New hydrological cycle



TRL: 4 → 6

Capacity: 0.1 m³/day (K recovery & N recovery)

Quantifiable target: first results 55% K recovery; 75% N recovery; 60% Ca recovery; 55% Mg recovery

Status/progress:

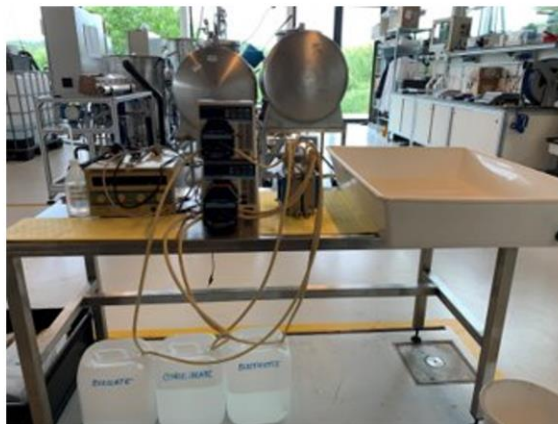
- Performance validation for Proof of Concept on laboratory scale being finalized.
- Detailed pilot design – completed
- Construction of pilot plant – completed & pilot plant - operational



CS2: Pictures/videos of the new technologies

Subtask: 1.4.1 Recovery of nutrients from greenhouse wastewater in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

Electrodialysis experiments



Lab experiments ongoing at KWR



Pilot experiments ongoing at Ghent University



Lab experiments to continue for remainder of project to support pilot.



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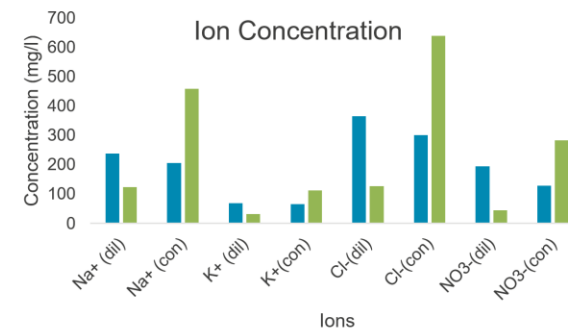
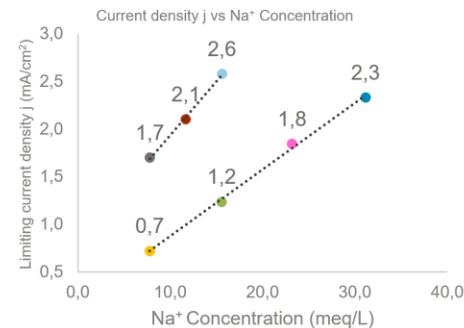




CS2: Results of the laboratory experiments

Subtask: 1.4.1 Recovery of nutrients from greenhouse wastewater in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

See also subtask 1.2.2: For the recovery of nutrients and their reuse, nutrient concentration and the removal of sodium is required. Thus, the described experiments in subtask 1.2.2 do also apply to this subtask 1.4.1.



12



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CS2: Operational procedures and methodologies

Subtask: 1.4.1 Recovery of nutrients from greenhouse wastewater in N. Prinsenland and Coöperatieve Tuinbouw Water Zuivering de Vlot

For the recovery of nutrients, the following treatment steps are being established:

- 1) Nutrient concentration
- 2) Efficiency in retaining nutrients (N, P, K) in the matrix and sodium removal
- 3) Optimal operational conditions and energy requirements

13



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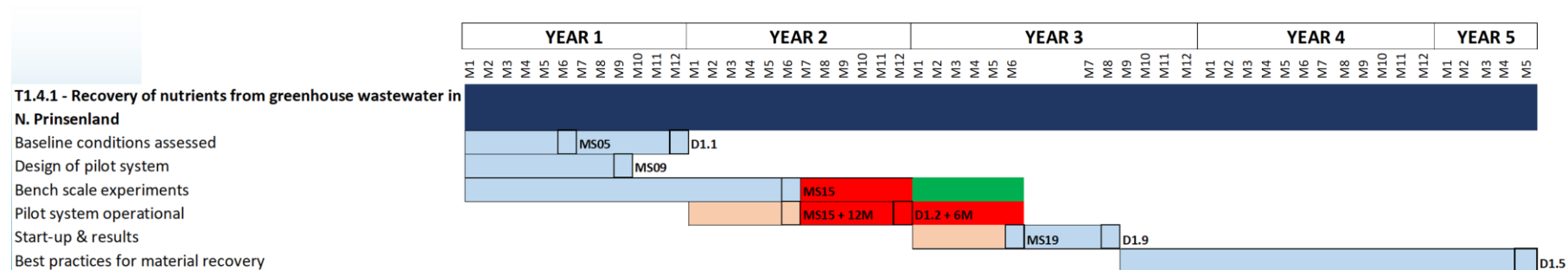


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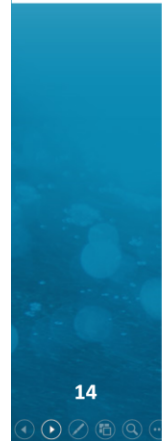


CS2: Subtask 1.4.1 – Timeline

Subtask: 1.4.1 Recovery of nutrients from greenhouse wastewater in N. Prinsenland and Coöperatieve Tuinbouw Water
Zuivering de Vlot



→ Pilot system operational since November 2022 (M30)



14



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Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity



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WATER SMART INDUSTRIAL SYMBIOSIS

CS2 Contacts

Tavishi.Guleria@kwrwater.nl

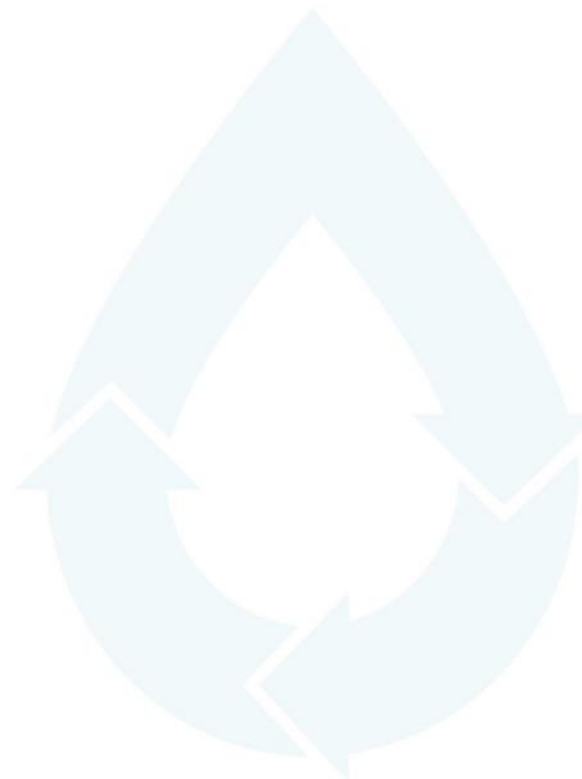
Joep.van.den.Broeke@kwrwater.nl



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2.3. CS3: Rosignano

Overview			D1.2: Operational demo cases in M24			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Expected to be operational [M]
3	1.2.3	Control system to avoid high chlorine concentrations	No pilot plant --> excluded from D1.2			
	1.4.2	Use of byproducts: pilot scale adsorption system	100%	100%	100%	Jul 22
		Use of byproducts: clari-flocculation pilot	100%	10%	0%	Jan 24
		AOP pilot system	60%	100%	0%	Jun 23



D1.2 Operational demo cases CS3 - Rosignano

UNIVPM, ARETUSA, WEST, CPTM





CS3: Rosignano

Lead partner (PPP site operator):



Other partners:

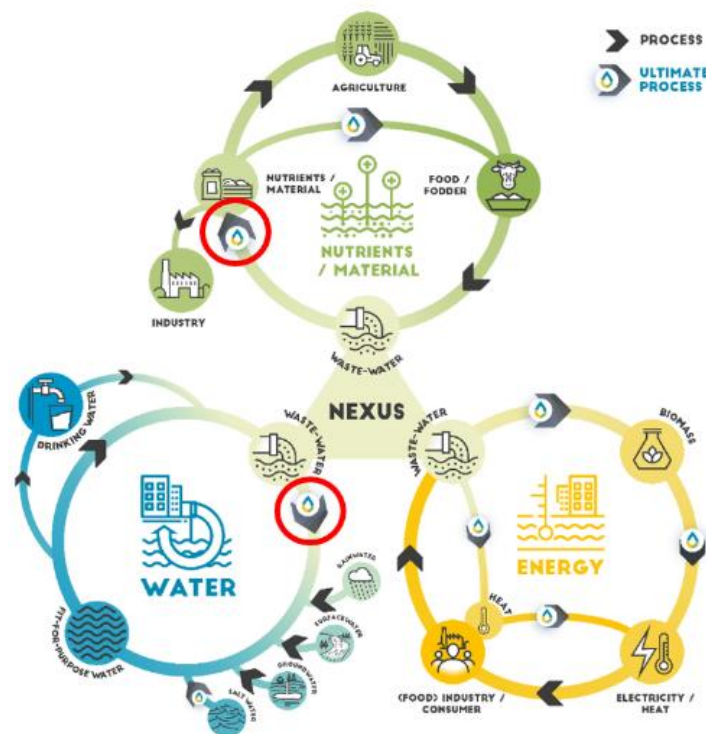


UNIVERSITÀ
POLITECNICA
DELLE MARCHE



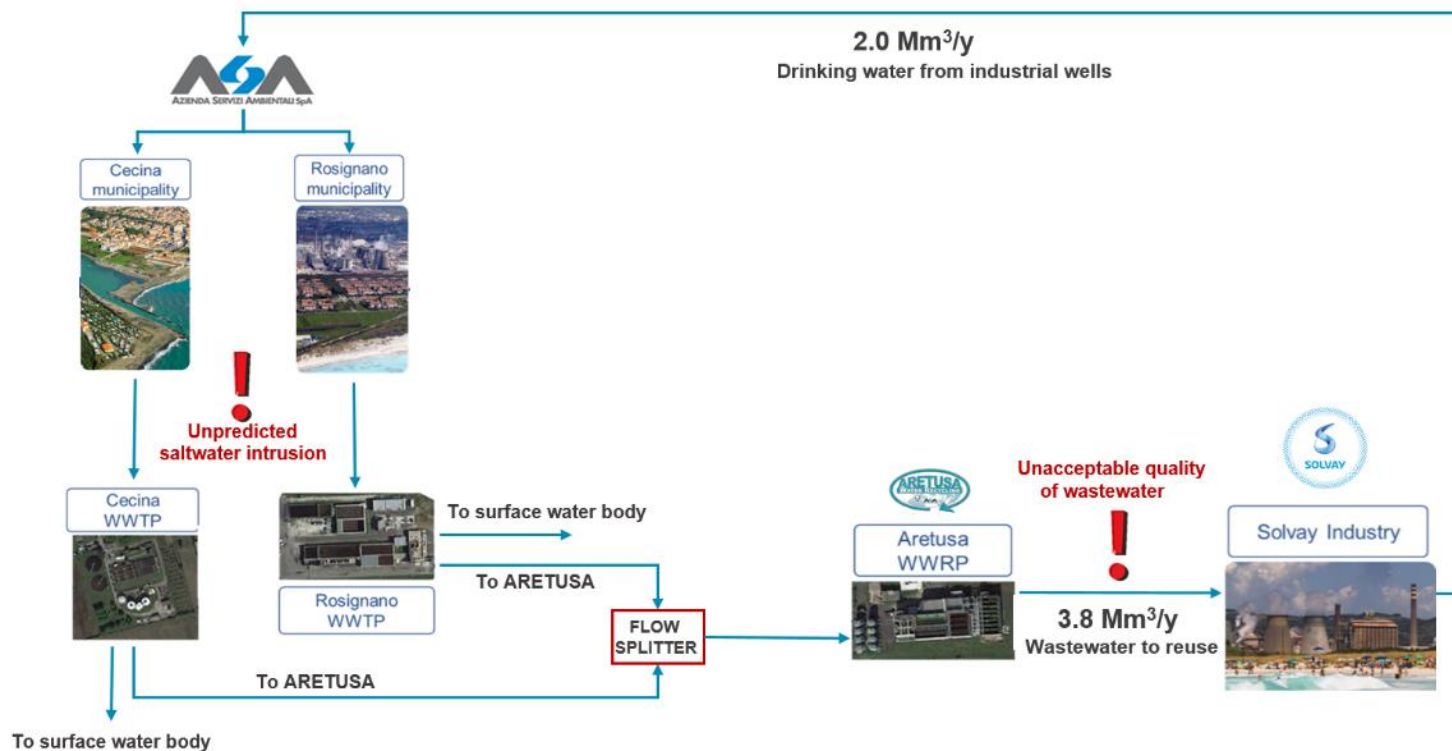
Consorzio Polo Tecnologico Magona

WEST
Systems





CS3: Situation before Ultimate



3



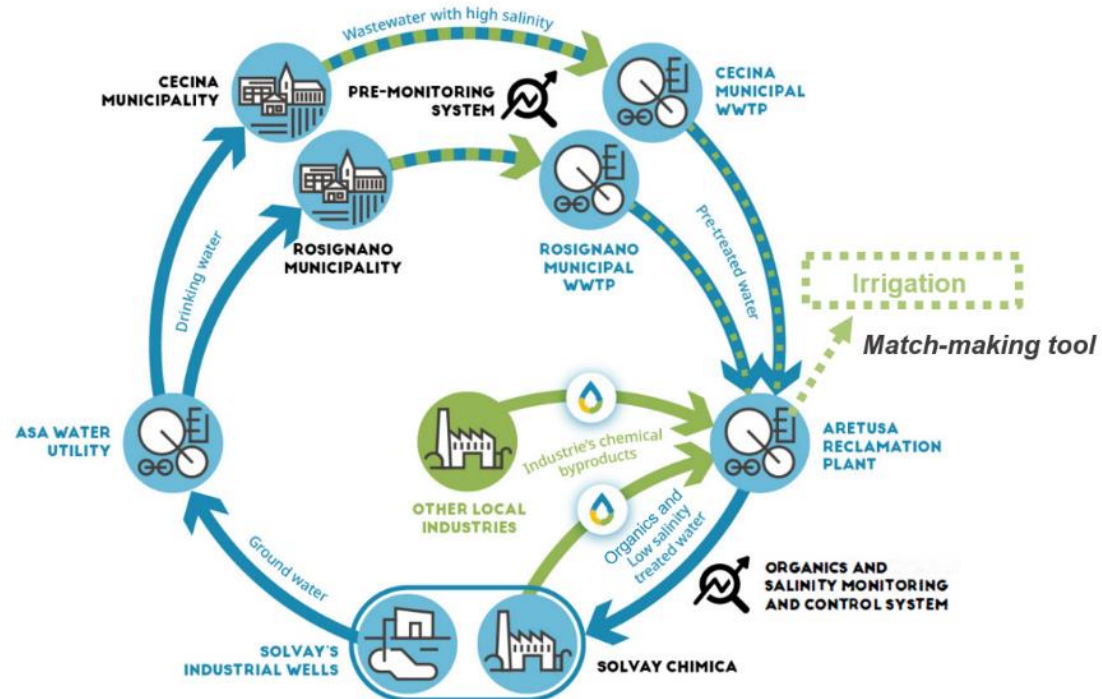
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CS3: Objectives of the Ultimate solutions



4



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CS3: Subtask 1.4.2 Status/progress

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

Baseline technology: No material reuse is in place so far

Ultimate solution to foster circular economy: Adsorption pilot with alternative GAC, and AOP pilot

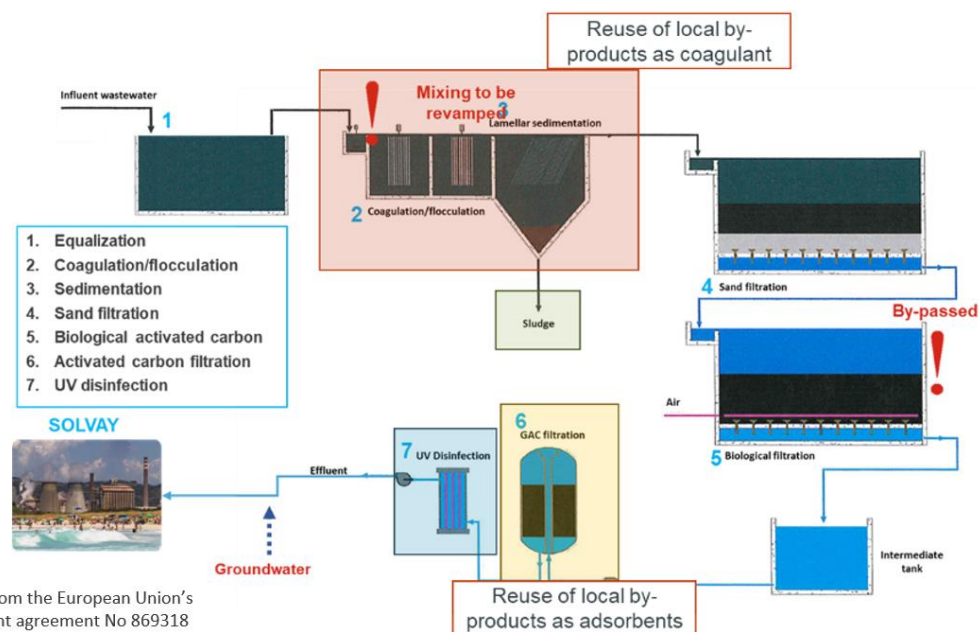
TRL: 4 → 7

Capacity: < 50 m³/h

Quantifiable targets: > 10% material recovery

Status/progress:

- Detailed design completed
- Adsorption pilot completed & in operation
- AOP pilot under construction
- Clari-flocculation plant under construction



5



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CS3: Results of the functional test

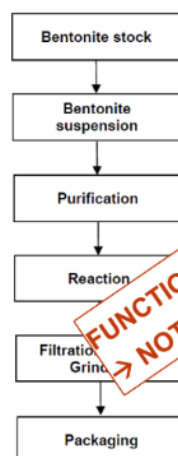
Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

MATERIALS (BY-PRODUCTS) CHARACTERIZATION

ORGANOCLAY (LAV1)



Laviosa Chimica Mineraria SpA extracts, process and distributes industrial mineral products, in particular bentonitic products and special 'modified' bentonitic products called "Organo-clay". From the necessary purification stages in the organic production process comes this 'grit' that is poor in bentonite but rich in zeolite.



**FUNCTIONAL TESTS SHOWED LOW PERFORMANCES
→ NOT FURTHER TEST OR SCALE-UP**

La composizione degli ossidi invece risulta come nella seguente tabella:

Fase	Quantità [%]
Zeoliti	33
Calcite	28
Plagioclasio	15
Quarzo	10
Mica	8
K-feldspato	6

Oxides composition

Analita	Quantità [%]
Na ₂ O	1,80
MgO	0,86
Al ₂ O ₃	11,75
SiO ₂	59,05
P ₂ O ₅	0,35
K ₂ O	2,76
CaO	9,85
TiO ₂	0,30
MnO	0,40
Fe ₂ O ₃	1,22

COMMERCIAL ACTIVATED CARBON (CA)

TES

CARBONE ATTIVO GRANULARE FA 300-SB

Origine	Antracite attivata con vapore d'acqua	
Granulometria, U.S. Mesh.	8 x 30	ASTM D 2862
	> 8 (2,36 mm)	5 % max.
	< 30 (0,60 mm)	4 % max.
Densità apparente, g/l	490 - 540	ASTM D 2854
Umidità all'imballaggio, %	< 2	ASTM D 2867
Durezza, %	> 95	ASTM D 3802
Indice di abrasione, %	> 90	AWWA B 604/74
Indice di Iodio, mg/g	> 950	ASTM D 4607
Indice di Blu di Metilene, mg /g	> 220	Spettrofotometrico





CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

MATERIALS (BY-PRODUCTS) CHARACTERIZATION: HYDROCHAR ACTIVATION

Physical activation – ATT1

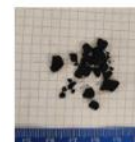
- Heating of the char pellets in a tubular oven up to 700°C (5°C/min) with N₂ purging.
- CO₂ flushing and isotherm for 2 hr.
- Cooling of the tubular furnace in N₂ purging.



47%
WEIGHT
LOSS

Chemical activation – ATT4

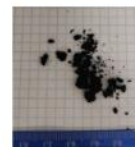
- Impregnation of char pellets in KOH aq. solution (KOH to char ratio: 1:1) at 60°C for 6 hr.
- Drying of the impregnated char at 105°C.
- Heating in a tubular oven up to 600°C (5°C/min), isotherm at 600°C for 1 hr and cooling (5°C/min) with N₂ purging.
- Washing with 1M HCl and demi water (up to pH 7).
- Drying at 105°C until constant weight.



55%
WEIGHT
LOSS

Chemical activation – ATT5-ATT6/7

- Mixing of the char pellets (previously grounded) with KOH in flakes (KOH to char ratio: 1:1).
- Heating in a tubular oven up to 600°C (5°C/min), isotherm at 600°C for 1 hr and cooling (5°C/min) with N₂ purging.
- Washing with 5M HCl and demi water (up to pH 7).
- Drying at 105°C until constant weight.



60%
WEIGHT
LOSS





CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

MATERIALS (BY-PRODUCTS) CHARACTERIZATION

	LAV1	HC	HC ATT1	HC ATT4	HC ATT5
F ⁻	< 0.1	54.2	0.2	0.4	< 0,1
Cl ⁻	2,5	44.4	62.5	26.4	1.7
NO ₃ ⁻	0.2	1.3	< 0,1	< 0,1	< 0,1
PO ₄ ³⁻	3.2	38.5	< 0,1	54.3	8.4
SO ₄ ²⁻	32.9	147.3	143.9	110.2	103.4
COD	81	4200	< 15	< 15	< 15

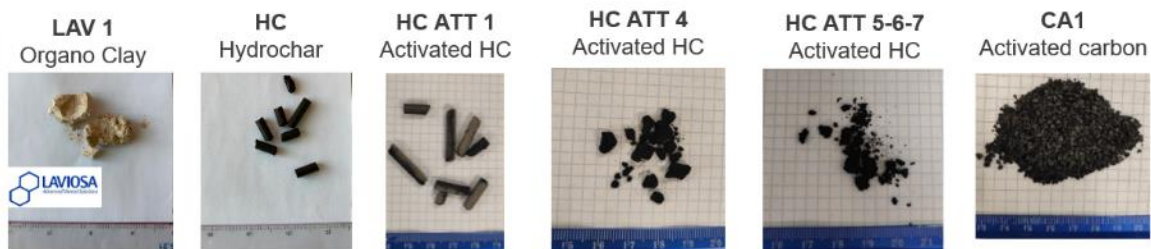


- ✓ RAW (NOT ACTIVATED) HYDROCHAR CONTAINS TAR → HIGH COD
- ✓ NEED OF PRE-TREATMENT (WASHING) OF RAW HYDROCHAR (NOT ACTIVATED)

	LAV1	HC ATT1	HC ATT4	HC ATT5	CA1
Specific surface area (m ² /g)	6	117	449	752	1100÷1150
Specific pore volume (cm ³ /g)	0.003	0.055	0.214	0.359	-
Average pore radius(Å)	50.23	13.61	15.16	16.08	-



- ✓ COMMERCIAL ACTIVATED CARBON (CA) WAS USED AS REFERENCE FOR THE ADSORPTION TESTS
- ✓ HIGH SURPHACE AREA DEVELOPED BY ACTIVATED HYDROCHAR



8



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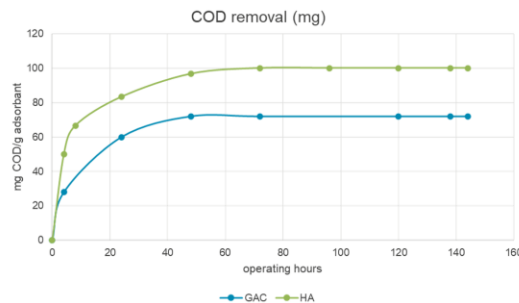
CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

ADSORPTION TESTS

Material tested: Activated Hydrochar (AH) and Commercial Granular Activated Carbon (GAC)

KINETIC AND ISOTHERM FOR COD ADSORPTION IN MUNICIPAL WASTEWATER



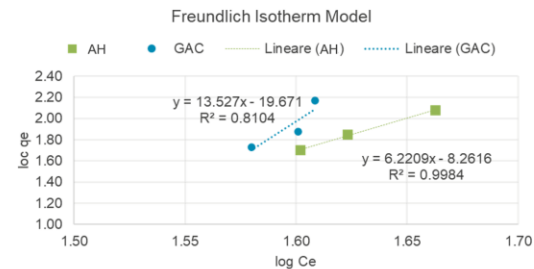
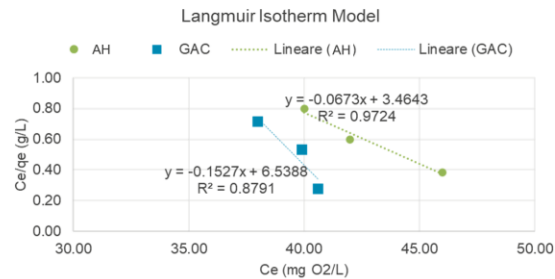
KINETIC	EQUATION	PARAMETERS	GAC	HTC
Pseudo-First Order	$\log(qe - qt) = \log(qe) - \frac{k_1 t}{2.303}$	k_1 min ⁻¹	0.164	0.148
		R ²	0.987	0.968
Pseudo-Second Order	$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{t}{qe}$	k_2 g/mg/min	0.003	0.002
		R ²	0.999	0.999

ISOTHERM	EQUATION	PARAMETERS	GAC	HTC
Freundlich	$\log qe = \log K_f + (1/n) \log Ce$	$1/n$	13.53	6.221
		K_f (mg/g)(L/mg) ^{1/n}	2.86E-09	2.58E-4
		R ²	0.810	0.998
Langmuir	$\frac{Ce}{qe} = \frac{1}{ab} + \frac{1}{a} Ce$	a mg/g	6.547	14.87
		b L/mg	0.023	0.019
		R ²	0.879	0.972

✓ AH has a higher % of COD removal in a shorter time: in the first 8 hours 60% of COD was removed with HA and 25% with GAC.

✓ Adsorption processes in both AH and GAC can be described by Pseudo Second Order kinetic model, while the Langmuir Isotherm model showed a better fit than the Freundlich model.

✓ 100 mg and 70 mg of COD was removed by AH and GAC respectively after 72 operating hours.





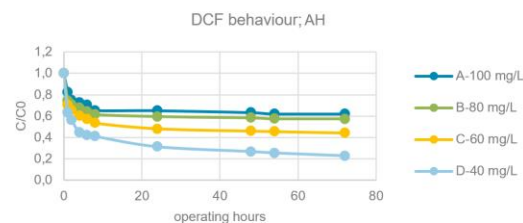
CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

ADSORPTION TESTS

Material tested: Activated Hydrochar (AH) and Commercial Granular Activated Carbon (GAC)

KINETIC WITH DICLOFENAC SOLUTION



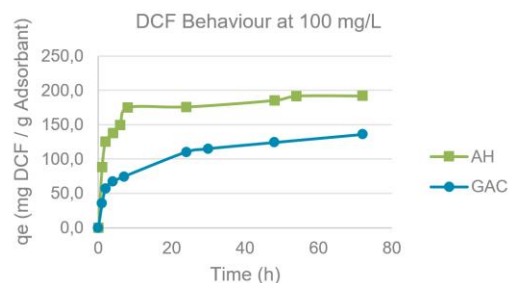
Experimental Setup for Batch Tests

✓ *AH showed higher adsorption capacity than conventional GAC.*

✓ *The adsorption equilibrium was reached, after 72 operating hours for HTC and after 144 hours for GAC*

ADSORBANT MATERIAL: GAC							
KINETICS	EQUATION	PARAMETERS	A	B	C	D	
Pseudo-first Order	$\log(qe - qt) = \log(qe) - \frac{k_1 t}{2.303}$	k1 min ⁻¹	0.06	0.07	0.07	0.07	
		R ²	0.93	0.96	0.97	0.97	
Pseudo-Second Order	$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{t}{qe}$	k2 g/mg/min	0.001	0.001	0.001	0.001	
		R ²	0.999	0.999	0.999	0.999	

ADSORBANT MATERIAL: AH							
KINETICS	EQUATION	PARAMETERS	A	B	C	D	
Pseudo-first Order	$\log(qe - qt) = \log(qe) - \frac{k_1 t}{2.303}$	k1 min ⁻¹	0.20	0.148	0.129	0.116	
		R ²	0.803	0.863	0.893	0.899	
Pseudo-Second Order	$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{t}{qe}$	k2 g/mg/min	0.003	0.005	0.003	0.003	
		R ²	0.999	0.999	0.999	0.999	



10



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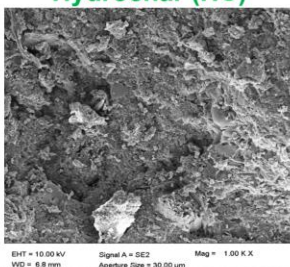
CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

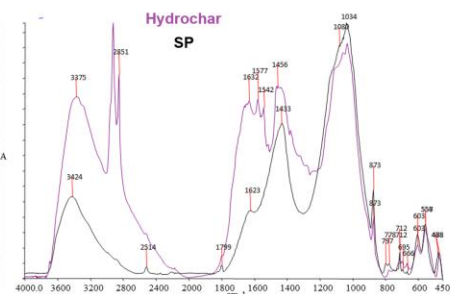
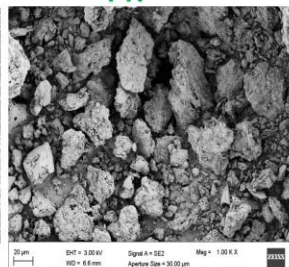
ADSORPTION TESTS

Material tested: Pyrolyzed Hydrochar (PH) and Commercial Granular Activated Carbon (GAC)

Hydrochar (HC)



PH



- ✓ Pyrolyzed hydrochar (PH) is much more chopped and less compact.
- ✓ Thermal process effectively eliminated the organic part (TAR) from the material.

MATERIALS		HC	AH	PH	GAC
BET surface area	m ² /g	ND	751.7	100.42	1100-1150
Maximum pore volume	cm ³ /g	ND	0.359	0.045	-
Median pore width	Å	ND	16.08	7.672	-

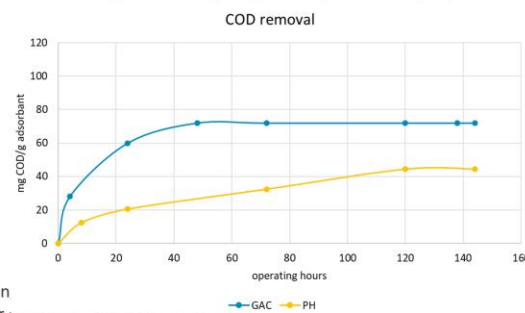
KINETIC WITH MUNICIPAL WASTEWATER

Adsorption on Activated Hydrochar (AH)



KINETIC	EQUATION	PARAMETERS	GAC	SP
Pseudo-First Order	$\log(q_e - qt) = \log(q_e) - \frac{k_1 t}{2.303}$	k_1 R^2	min^{-1} 0.164 0.987	0.0452 0.90
Pseudo-Second Order	$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{t}{k_2 q_e}$	k_2 R^2	g/mg/min 0.003 0.999	0.0093 0.90

Adsorption on Pyrolyzed Hydrochar (PH)



- ✓ PH has a slower adsorption kinetic of COD compared with AH and GAC. Lower COD removal was observed.



CS3: Results of the functional test

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

MATERIALS (BY-PRODUCTS) CHARACTERIZATION

- ✓ “Precotto”: granulated limestone rocks only partially calcinated and slacked, with a declared content of Ca(OH)_2 of about 9%.
- ✓ Na_2CO_3 “Soda Solvay® Light” product that resulted to be out of specification.

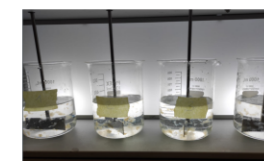


Solvay Chimica Italia
SpA by-products tested



SOFTENING/COAGULATION/FLOCCULATION TESTS

SUBSTRATE	SOFT. AGENT	pH	COD Removal (%)	Mg Removal (%)	Ca Removal (%)
Influent municipal wastewater	Commercial SODA 1M	8.5-10		0	< 53
Influent municipal wastewater	Soda Solvay	8.5-10		0	44-80
Influent municipal wastewater	Precotto	8.5-10		4-8	< 35
Influent municipal wastewater	Precotto	8-9.5	49-58	0	17-24
Effluent wastewater	Precotto	8-9.5	25-40	7-19	0
Effluent wastewater	Soda Solvay	8-9.5	< 10	0	7-45
Aretusa wastewater	Precotto	9-9.5	7-47	9-11.4	5.7-9
Aretusa wastewater	Soda Solvay	9-9.5	47-73	0-8.4	9-24
Aretusa wastewater	Precotto and Soda Solvay	9-9.5	80-87	4-6.2	10-24



- ✓ Solvay by-products proved to be successful in reducing COD and, even if with lower performances, Magnesium and Calcium as well
- ✓ Final test are going to be performed to optimize the dosage

12





CS3: Pictures/videos of the new technologies

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

ADSORPTION PILOT SYSTEM CONSTRUCTION



13



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CS3: Pictures/videos of the new technologies

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

ADSORPTION PILOT SYSTEM START-UP

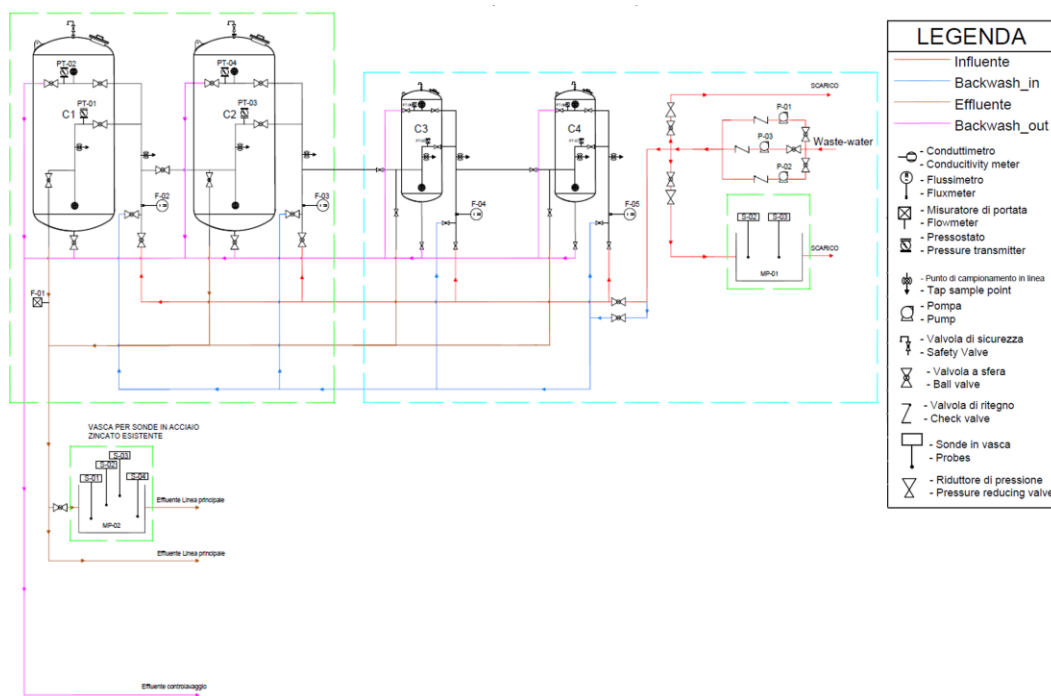




CS3: Operational procedures and methodologies

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

ADSORPTION PILOT SYSTEM



- ✓ Pilot plant has been designed to **use adsorption columns both in series and in parallel**
- ✓ The pilot is able to **work with different flow rates** in order to optimize the operation of bigger and smaller columns.
- ✓ **Pressure** in all the columns will be **monitored online** to check when it is necessary to proceed with **back-washing operations that will be carried out with a counter-current water flow**.
- ✓ **Conductivity, pH and COD** (UV/Vis and fluorescence) **will be monitored** at the exit of the pilot. COD will be monitored also in the incoming flow.
- ✓ **All sensors, pressure transmitters and pumps will be connected to the electrical cabinet and data will be available online**
- ✓ The pilot will be firstly installed and operated at the pilot hall of UNIVPM and than will be transported and installed at ARETUSA site

15



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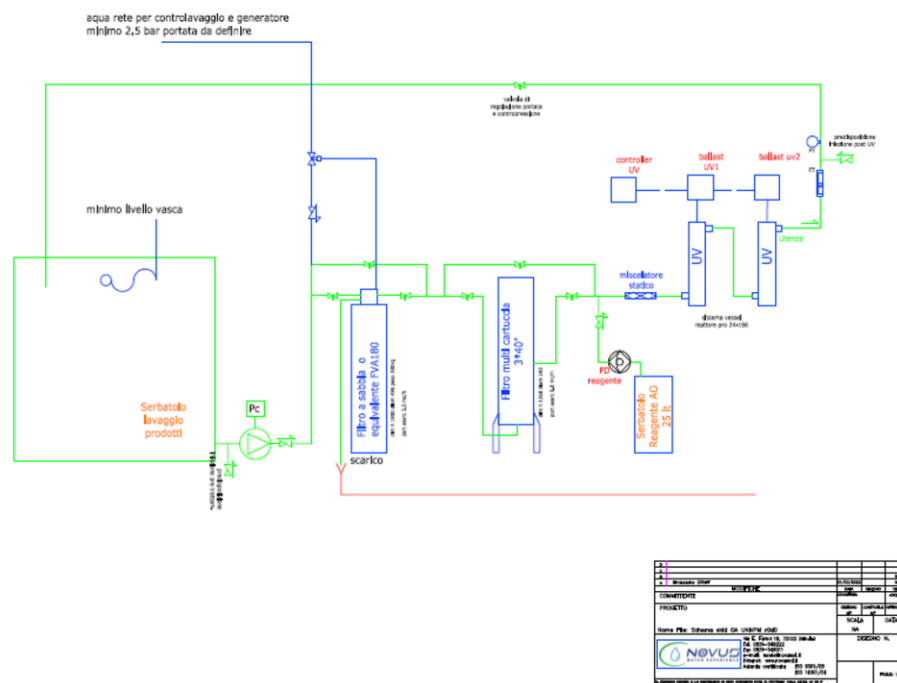
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CS3: Operational procedures and methodologies

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

AOP PILOT SYSTEM



SKID EQUIPPED WITH:

- ✓ pressurization pump: 60 l/min, 1.9 bar
- ✓ sand filter: 3.5 mc/h 250 kg of sand
- ✓ multi-cartridge filter
- ✓ electromagnetic dosing pump
- ✓ static mixer
- ✓ Viqua PRO 24-180mJ UV system
- ✓ electrical panel

Pilot plant under construction!

16



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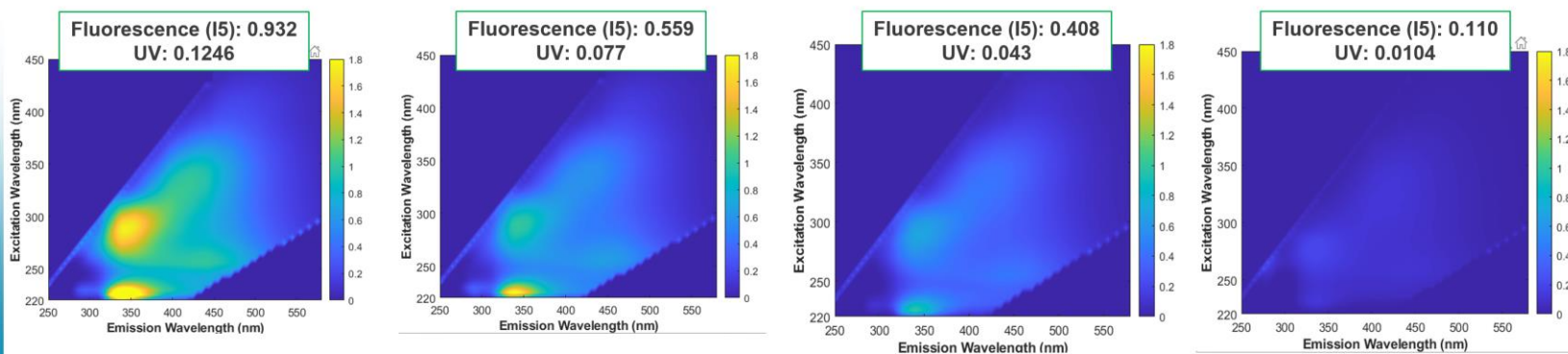
CS3: First results

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano

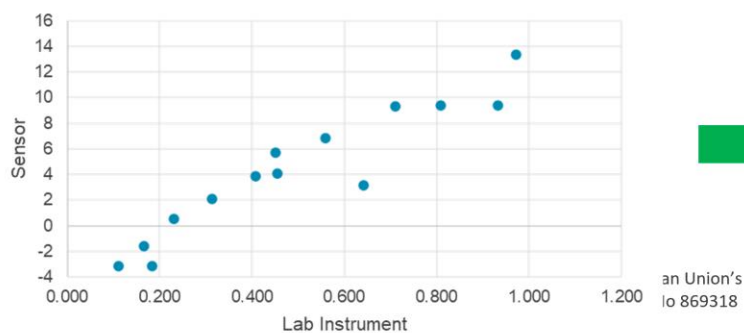
ADSORPTION PILOT SYSTEM

INFLUENT

EFFLUENT



FLUORESCENCE



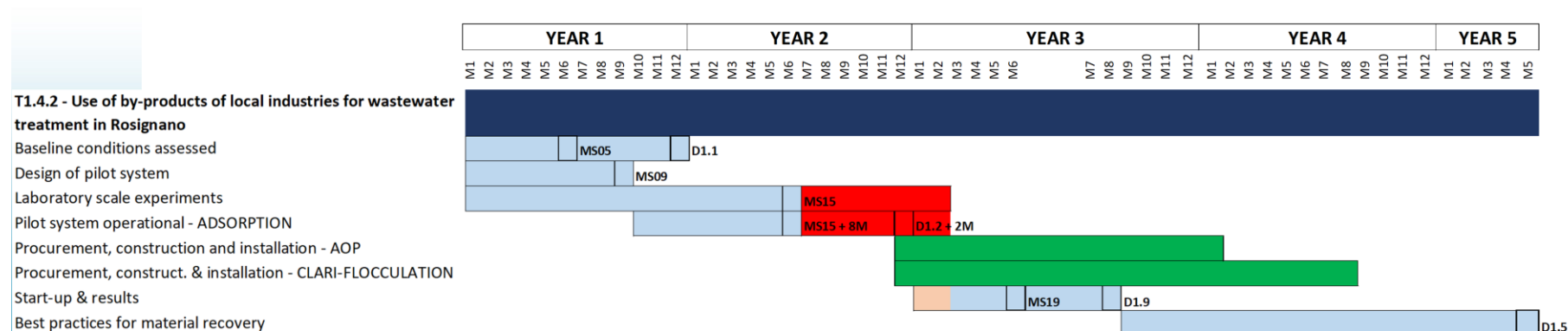
Calibration of the fluorescence sensor showed promising results!





CS3: Subtask 1.4.2 – Timeline

Subtask: 1.4.2 Use of by-products of local industries for wastewater treatment in Rosignano



- Pilot system is operational since July 2022 (M26)
- Two additional pilot systems will be implemented having a different timeline
- Still enough time to complete the pilot experiments
- Additional pilots will be presented in D1.5

Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity

18



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WATER SMART INDUSTRIAL SYMBIOSIS

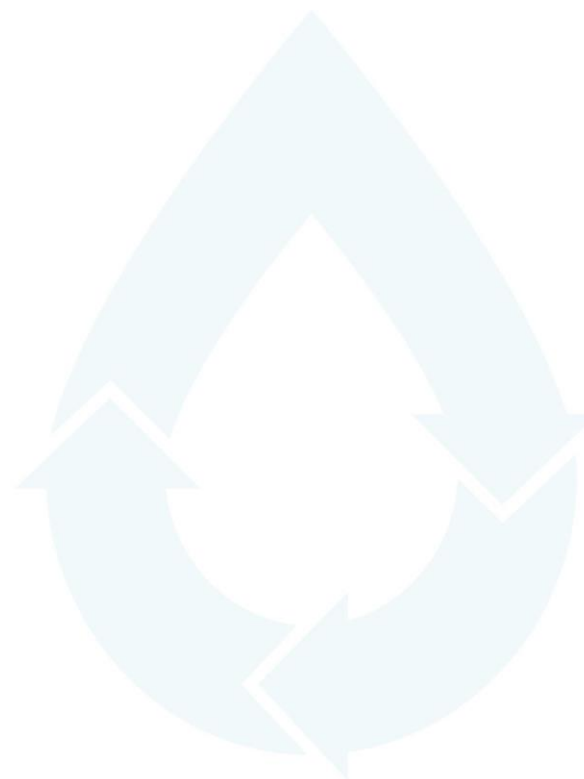
CS3 Contacts

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2.4. CS4: Nafplio

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
4	1.2.4	Water recovery: filtration, AOP, SBP	100%	100%	100%	May 22
	1.4.3	Recovery of antioxidants: adsorption/extraction	100%	100%	100%	Nov 22



D1.2 Operational demo cases

CS4 - Nafplio

Greener than Green, Alberta, NTUA



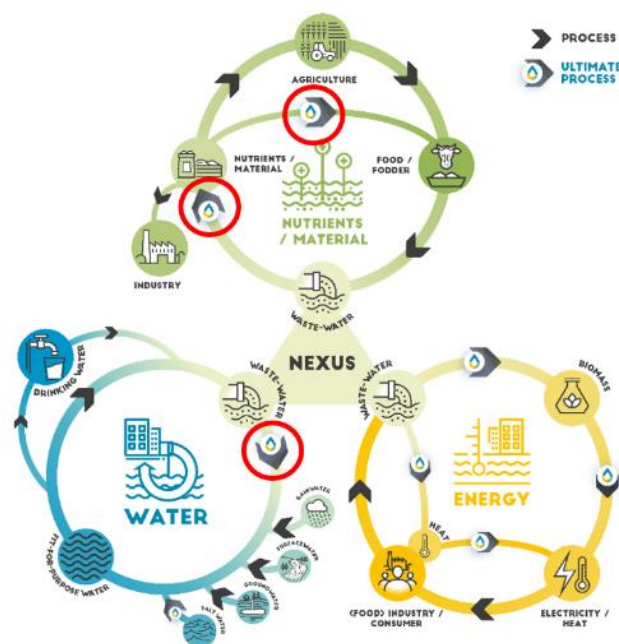


CS4: Nafplio

Lead partner:



Other partners:



2



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CS4: Situation before Ultimate

Argolida area:

- increasing water demand for irrigation
- high-water consumption of the fruit processing industry
- great pressure on regional aquifer

Alberta S.A has a primary treatment unit of about 20 m³/h capacity:

- high production periods (Nov.-Mar. & Aug.-Oct.): 3500 m³ WW/d
- other months: 500 m³ WW/d
- treatment unit consists of a series of tank:

Raw wastewater tank → Rotostrainer → Less solids tank → equalization/ homogeneous tank → Neutralization tank → Pre Sedimentation tank → Aeration tank → Flocculation tank → Final sedimentation tank → Final tank of treated water → Central treatment unit of local water authority (DEYARM)

Aim of the Ultimate solutions (after the implementation of the additional pilot wastewater treatment process):

- to achieve lower organic burden in the final effluent,
- compliant to limits specified by the local water management authority
 - either for disposal to the local final treatment unit,
 - either for irrigation
 - or for reuse in the production procedure of Alberta S.A.

3

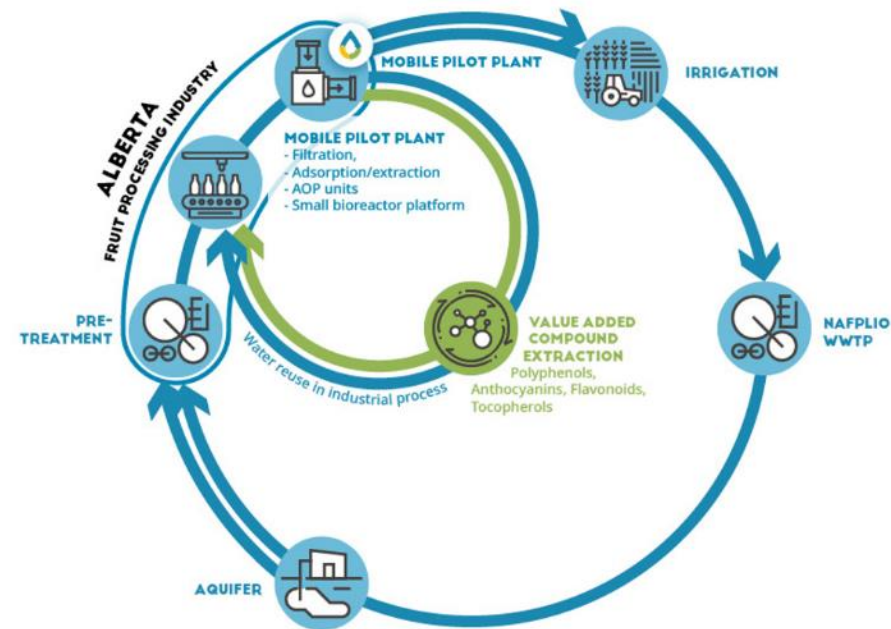


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CS4: Objectives of the Ultimate solutions



4



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CS4: Objectives of the Ultimate solutions

Ultimate aims to address the various issues involved in fresh-water management and reduce wastewater disposal cost. Thus, different techniques are to be implemented to guarantee a sustainable management of the end-of-the-pipe wastewater effluents derived from the food industry, and also to prevent the losses of inorganic and organic pollutants to the environment, making it easier to recycle/reuse the purified water.

The activities in ULTIMATE target both the recovery of various inorganic and organic contaminants from the processing water and the reuse of the purified water. In Alberta's fruit processing plant, a mobile pilot plant will demonstrate a hybrid adsorption / SubCritical Water Extraction (SCWE) process to extract high value-added compounds, such as antioxidants from the wastewater. Residual wastewater will be treated in pilot-scale by an AOP before polishing in an on-site Small Bioreactor Platform (SBP) for reuse in irrigation or discharge into the municipal WWTP to reduce operational costs. The extracted compounds will be assessed for their use by Alberta making "fortified juice" with antioxidant properties, increasing the value of their product, but also by selling the extract to the food-supplement sector.

5



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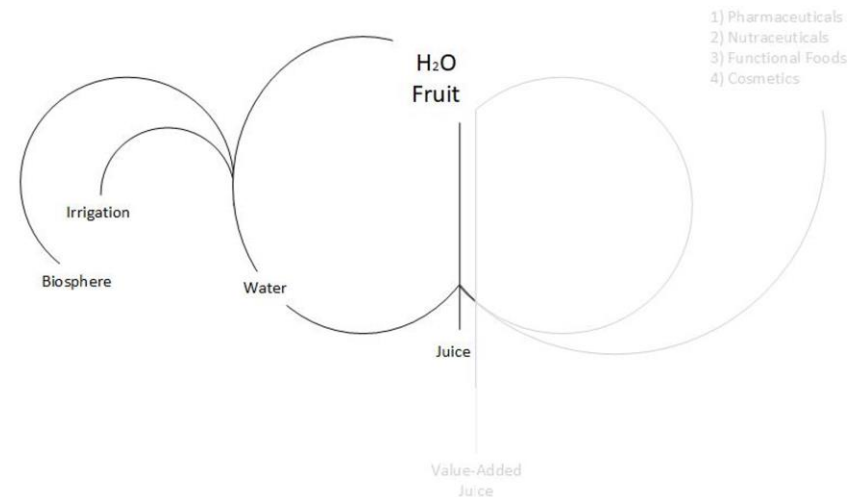


CS4: Subtask 1.2.4 Status/progress

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

Baseline technology: no water reuse so far

Ultimate solution to foster circular economy:



TRL: 5→7

Capacity: 10 m³/d

Quantifiable target: Ambition beyond the project: 100% water reuse for irrigation; >90% reduction of freshwater through water reuse

Status/progress:

- detailed design completed
- The unit has been installed in Nafplio and is operational

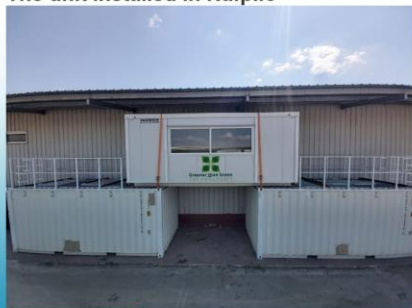




CS4: Pictures/videos of the new technologies

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

The unit installed in Nafplio



Dosing pumps



TOC analyzer



Sensors



Unit installation video



SBP capsules



7



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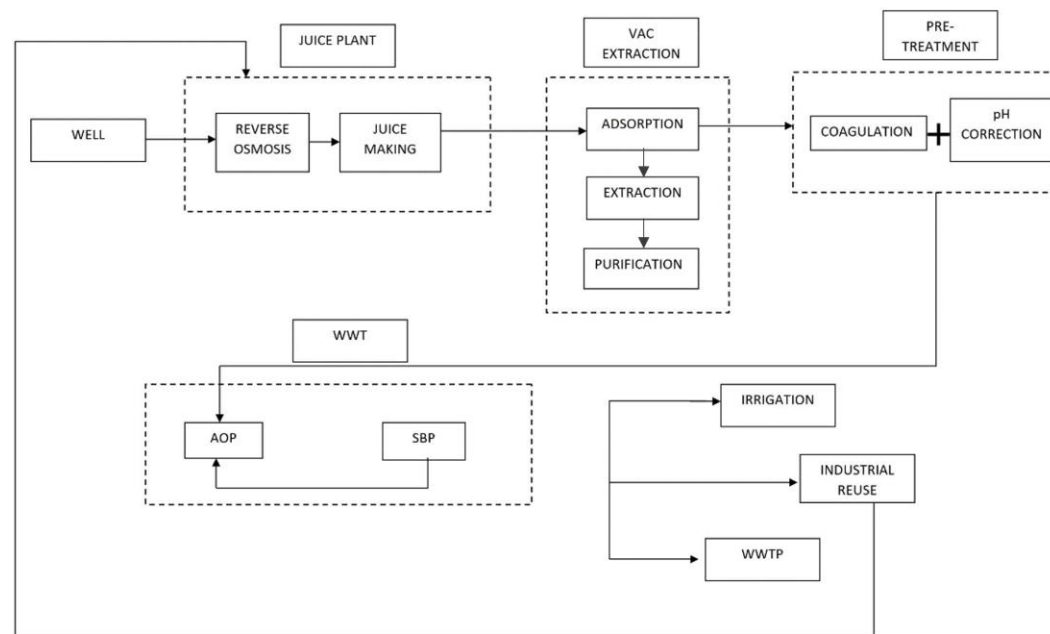


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CS4: Operational procedures and methodologies

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio



8



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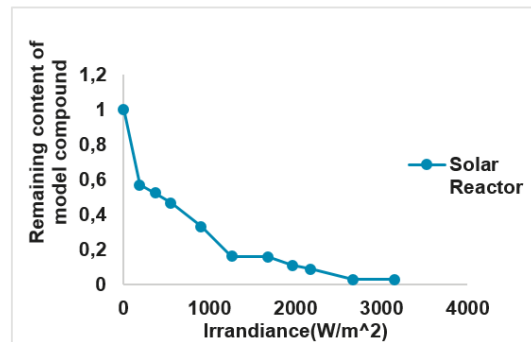


CS4: Results of laboratory experiments

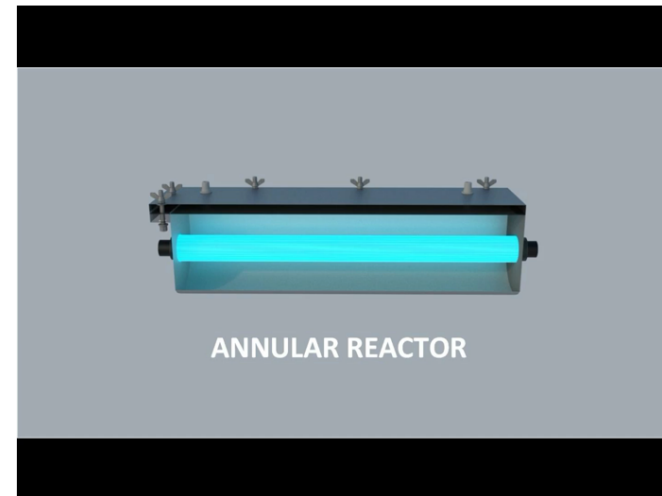
Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

Advanced Oxidation Process (AOP)

- **GtG has developed 2 AOP reactors**
- **Annular reactor:** a tube with a high intensity UV lamp where the wastewater flows through
- **Solar reactor:** made of quartz glass tubes and performs under solar light. It has been proven to effectively degrade our model compound



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Solar reactor: low OpEx as there are no energy costs, but to achieve the contact time needed for degradation you need either huge area of glasses either extremely low flow, these are obstacles for a pilot unit

Annular reactor: operates with UV lamps and shorter contact time is needed

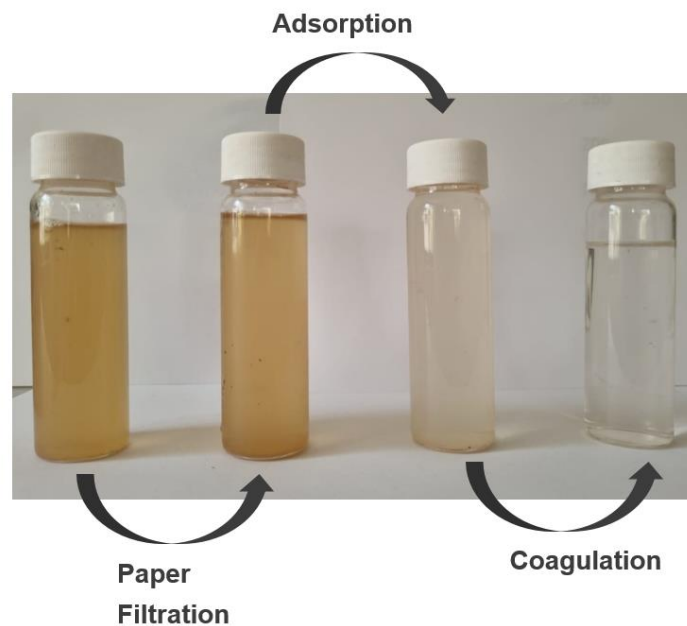




CS4: Results of laboratory experiments

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

Pretreatment steps- Orange by-product



	TOC (mg/L)
Orange by-product	1950
Filtrate (paper filtration)	1810
Solution after adsorption	1400
Coagulation supernatant	1005





CS4: Results of laboratory experiments

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

Results of the individual technologies

- Coagulant effectively removes TSS
- The adsorption of VAC is more efficient if it goes prior to any chemical process → Minor change in our initial design





CS4: Pictures of the pilot units

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio

Adsorption of polyphenols



AOP: Annular reactor



AOP: solar reactor



12



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CS4: Subtask 1.2.4 – Timeline

Subtask: 1.2.4 Reuse of fruit processing wastewater in Nafplio



T1.2.4 - Reuse of fruit processing wastewater in Nafplio

Baseline conditions assessed

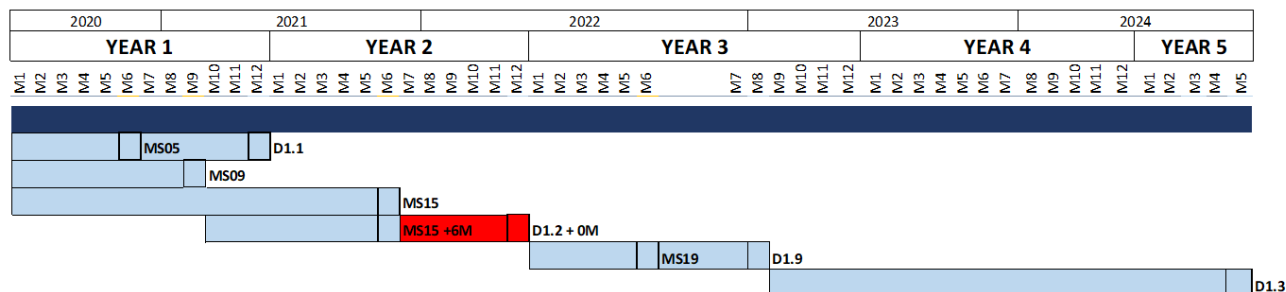
Design of pilot system

Laboratory experiments

Pilot system operational (Filtration, AOP, SBP)

Start-up & results

Best practices for water recycling



→ Pilot system (filtration, AOP and SBP) is operational since M24 (May 2022)



Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity



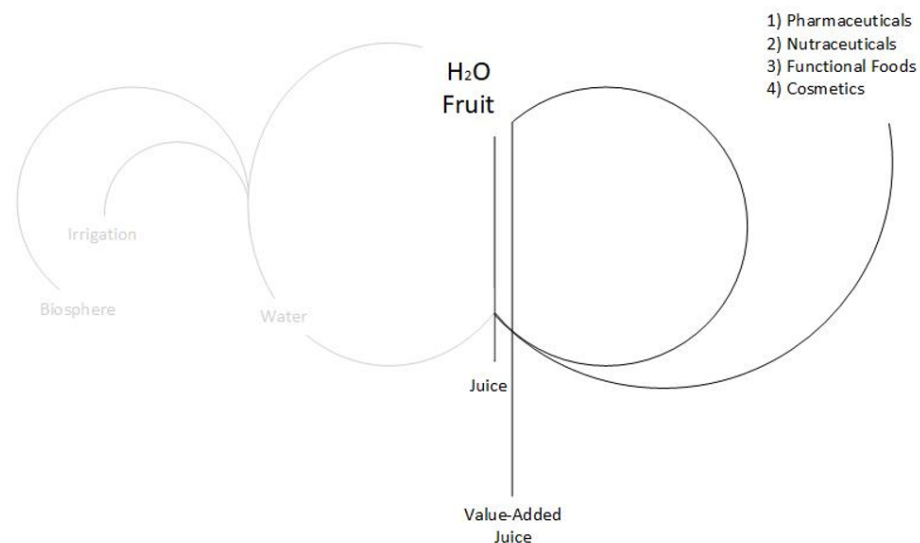


CS4: Subtask 1.4.3 Status/progress

Subtask: **1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio**

Baseline technology: **No recovery**

Ultimate solution to foster circular economy:



TRL: **5 → 7**

Capacity: **10 m³/d**

Quantifiable target: **Recovery of polyphenols: 50-70%**

Status/progress:

- **Lab scale experiments completed**
- **Pilot unit constructed and in operation**





CS4: Pictures/videos of the new technologies

Subtask: 1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio

Lab scale – Dynamic adsorption



Static adsorption



15



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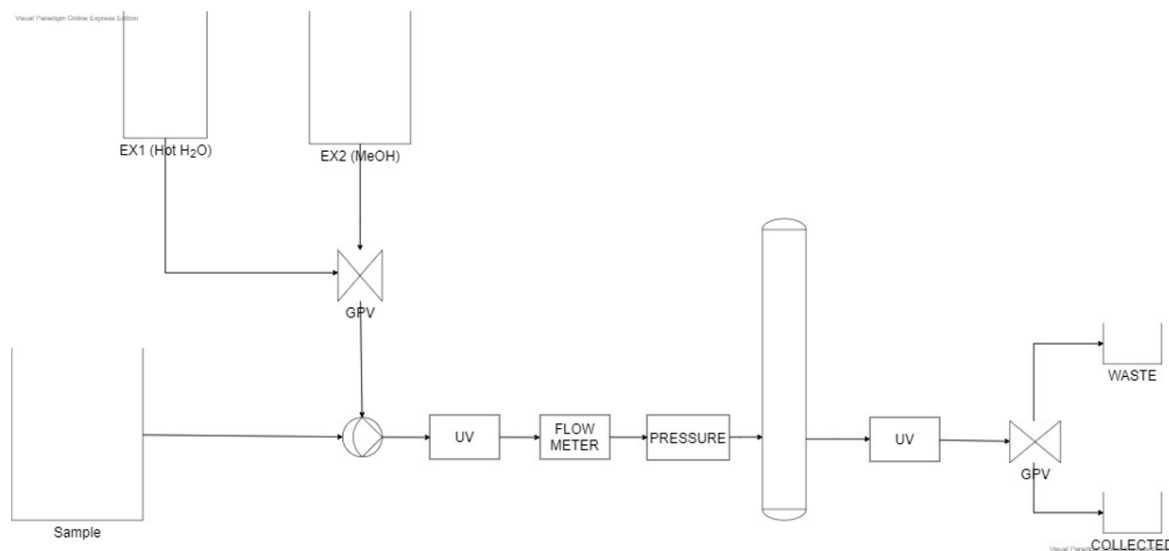


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CS4: Operational procedures and methodologies

Subtask: 1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio



16



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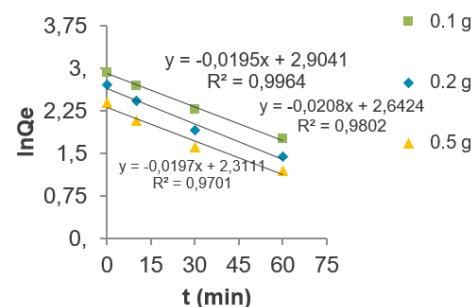
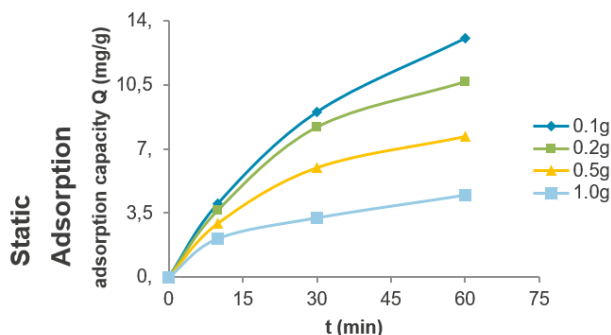


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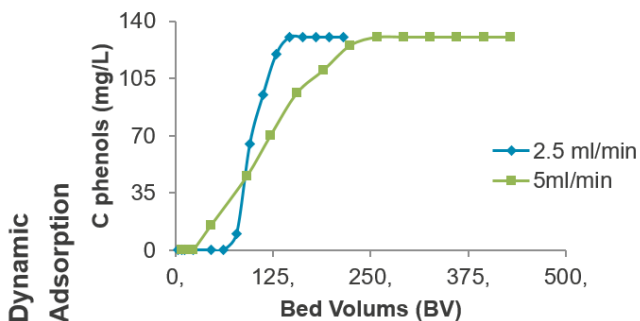


CS4: Results of the laboratory experiments

Subtask: 1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio



maximum adsorptive capacity (Q): 23 g of polyphenols per kg of resin for the FPX 66 resin



The breakthrough curves showed that 1.7 m³ of wastewater can be treated per kg of resin per 10 cycles





CS4: Results of the laboratory experiments

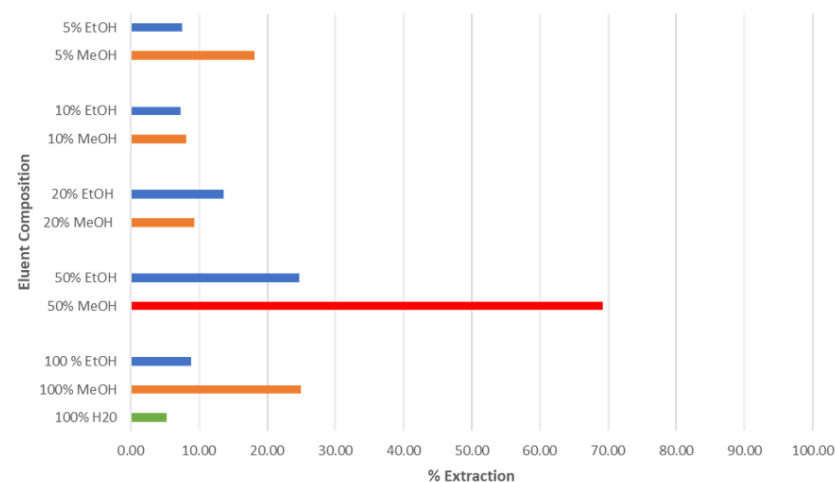
Subtask: **1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio**

- Static extraction experiments were performed employing hot water and organic solvents
- Water-methanol mixture (50:50 b.v.) yielded **69% polyphenols recovery**

• Currently working on dynamic extraction experiments,

• Aiming to optimise:

- experimental conditions and
- solvent recovery and reuse strategy





CS4: Pictures of the pilot units

Subtask: **1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio**

Adsorption of polyphenols



Supercritical water extraction



19



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CS4: Subtask 1.4.3 – Timeline

Subtask: **1.4.3 Recovery of high-added-value compounds (antioxidants) in Nafplio**



T1.4.3 - Recovery of high added-value compounds (antioxidants) in Nafplio

Baseline conditions assessed

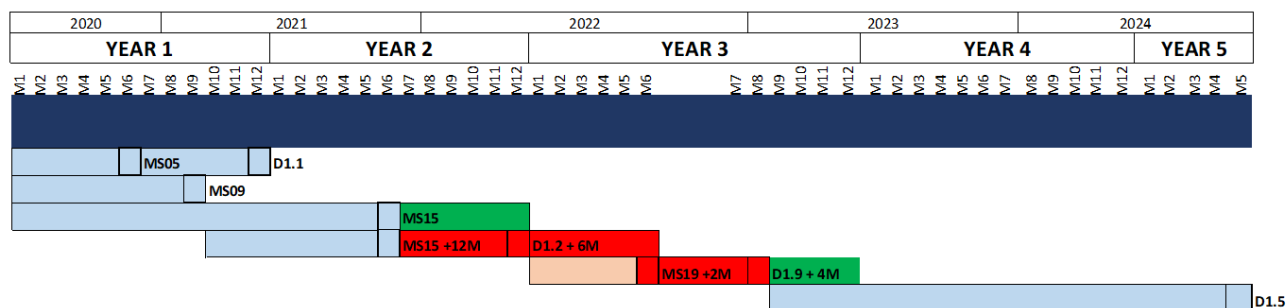
Design of pilot system

Laboratory experiments

Pilot system operational

Start-up & results

Best practices for material recovery



→ Pilot system operational since November 2022 (M30)



Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity

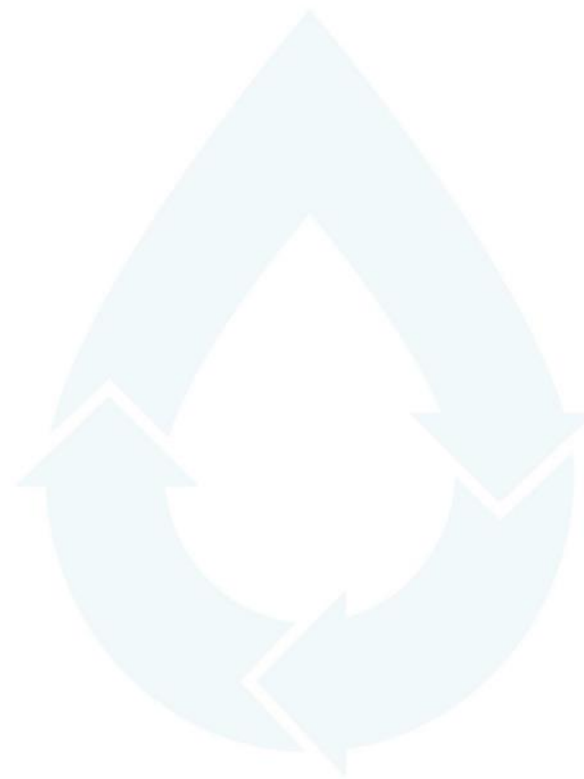




CS4 Contacts

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2.5. CS5: Lleida

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
5	1.2.5	Monitoring system for fouling in AnMBR (NF + RO) + (AOP + UV)	No pilot plant --> excluded from D1.2			
	1.3.2	AnMBR	100%	100%	100%	Jan./Jun. 22
		Pilot ELSAR	100%	100%	100%	Dec. 22
		Full-scale ELSAR		50%	0%	Sep 23
		SOFC		100%	100%	Dec. 22
	1.4.4	Concept study: recovery of nutrients	No pilot plant --> excluded from D1.2			
		Solar-driven hydrothermal carbonisation demo plant	100%	100%	100%	May 22



D1.2 Operational demo cases CS5 Lleida

AQUALIA, PENTAIR





CS5: LLeida

Lead partner:



Other partners:



2



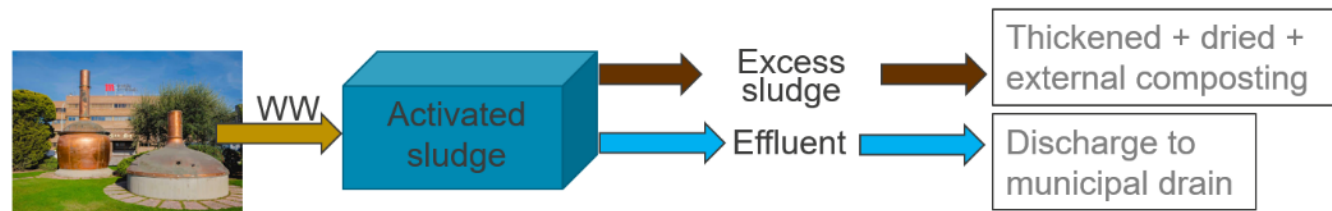
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CS5: Situation before Ultimate



3



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CS5: Objectives of the Ultimate solutions



4



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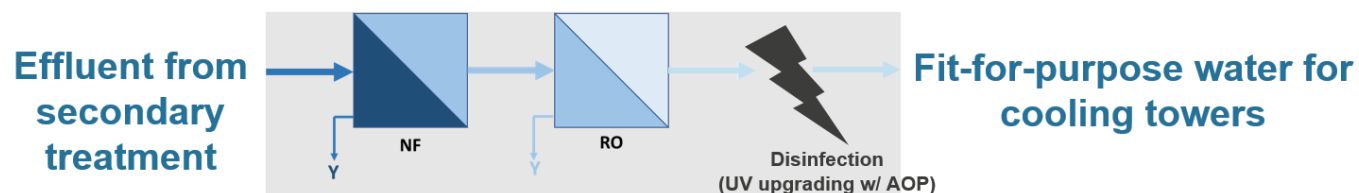


CS5: Subtask 1.2.5 Status/progress

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Baseline technology: no water reuse so far (only wastewater treatment with activated sludge process and subsequent discharge to the municipal drain)

Ultimate solution to foster circular economy: membrane-based technologies, disruptive disinfection/AOP technologies



TRL: 7 → 9

Capacity: 50 m³/d

Quantifiable target: 4200-4600 m³/a for cooling towers; 10-15% reduction of freshwater via reuse of treated water

Status/progress:

- Detailed design completed
- UF & RO: operational
- AOP & UV: operational



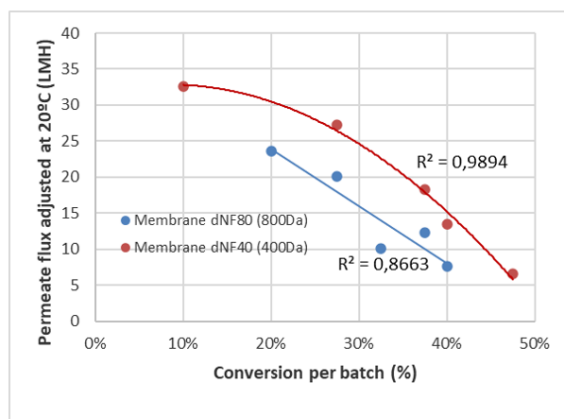


CS5: Results of laboratory experiments

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Conclusions from previous lab-scale tests:

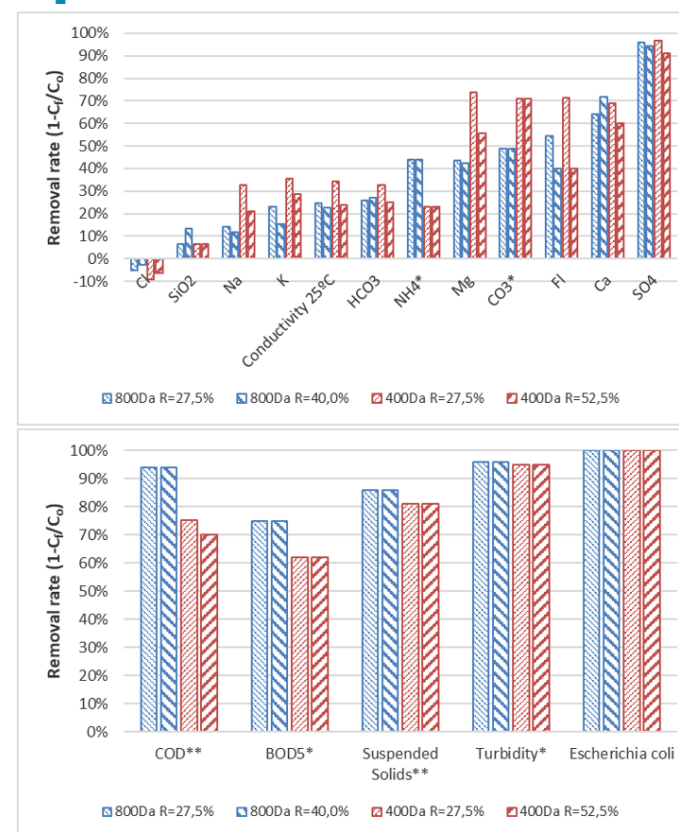
- NF is a valid technology for achievement of regulatory requirements, but for salinity removal a RO step is needed.
- 800Da is an enough membrane cut-off.
- Conversion should be kept as lower as possible to optimize filtration performance.



6



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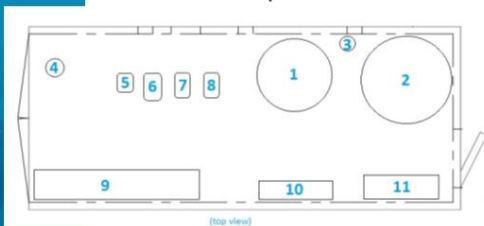
CS5: Pictures of NF & RO pilot system

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Nanofiltration demo plant.

Composed by:

1. Feed tank
2. Permeate tank
3. Amiad strainer
4. Membrane module
5. CIP circulation pump
6. Circulation pump
7. Feed pump
8. Backwash pump
9. Chemical cabinets
10. Panel PC
11. Compressor



Dimensions:
6,0m x 2,4m x 2,4m

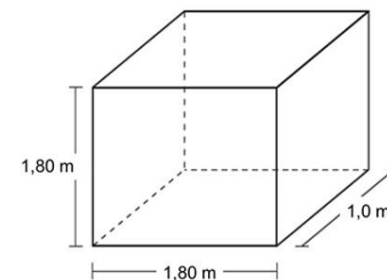


**Reverse osmosis
demo plant (1st trials)**

Composed by:

- Electrical cabinet
- 1 buffer tank
- 1 pressure vessel (2,5" membrane)
- 1 fabric filter
- 2 feeding pumps
- Several rotameters and manometers

Dimensions:



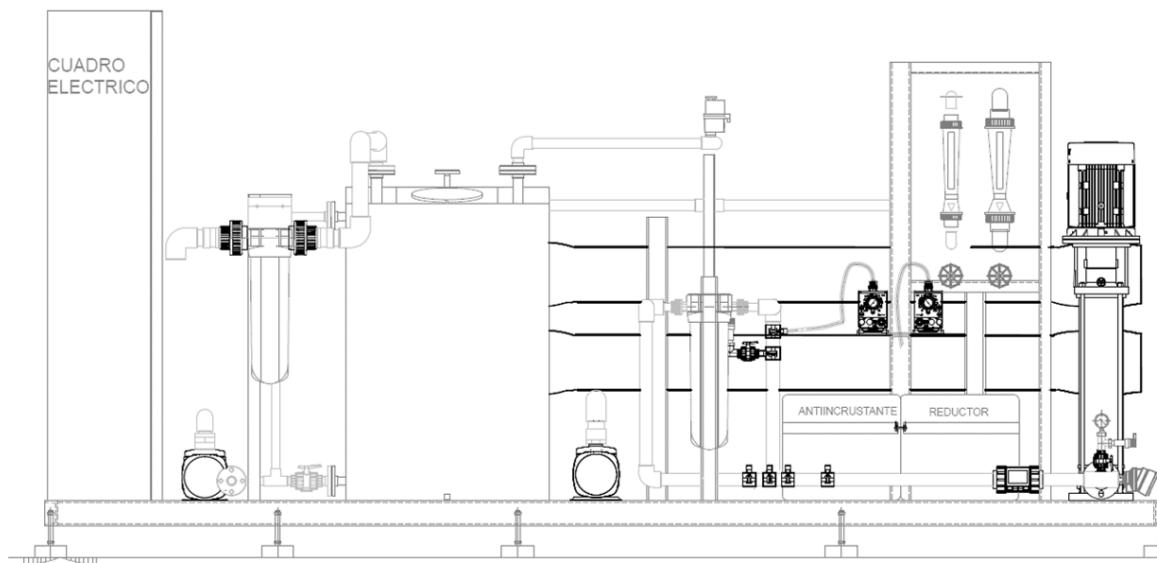


CS5: Pictures of NF & RO pilot system

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Reverse osmosis demo plant (2nd trials)

Due to water production limitations, a second larger demo-scale RO plant has been operated in Ultimate



Composed by:

- Electrical cabinet
- 1 buffer tank
- 2 RO spiral membranes
- 1 feeding pump
- 1 low-pressure pump
- 1 high-pressure pump
- 1 fabric filter
- 2 dosing pumps and tanks for dosing anti-scaling and disinfectant
- Continuous monitoring of conductivity, pH, flow
- Several rotameters and manometers

Dimensions:

- LxWxH: 4,5m x 1,4m x 2,1m

8



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CS5: Pictures of NF & RO pilot system

Subtask: 1.2.5 Reuse of brewery wastewater as process water



Nanofiltration demo plant



Reverse osmosis demo plant

9



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CS5: Operational procedures and methodologies

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Analytical plan

PARAMETER	INPUT WATER		OUTPUT NF / INPUT RO		OUTPUT RO or BLENDED REGENERATED WATER	
	Motivation	Frecuence	Motivation	Frecuence	Motivation	Frecuence
<i>"Legionella" sp</i>	Performance NF	Weekly	Performance RO and NF	Weekly	RD 1620/2007 (absence)	Weekly
Nematode eggs	Performance NF	Weekly	Performance RO and NF	Weekly	RD 1620/2007 (<1 unit/10L)	Weekly
<i>"Escherichia coli"</i>	Performance NF	Weekly	Performance RO and NF	Weekly	RD 1620/2007 (absence)	Weekly
Suspended solids	Performance NF	Weekly	Performance RO and NF/ requirement RO	Weekly	RD 1620/2007 (<5 mg/L)	Weekly
Turbidity	Performance NF	Weekly	Performance RO and NF/ requirement RO	Weekly	RD 1620/2007 (< 1NTU)	Weekly
Conductivity @ 25°C	Performance NF	Weekly	Performance RO and NF	Weekly	Required by cooling tower	Weekly
BOD5	Performance NF	Weekly	Performance RO and NF/ requirement RO	Weekly	UE 2020/741	Weekly
COD	Performance NF	Weekly	Rendimiento NF	Weekly	-	Weekly
pH	Requirement NF	Weekly	Required by RO step	Weekly	Required by cooling tower	Weekly
Alcalinity	-	0	-	0	Required by cooling tower	Weekly
Hardness	-	0	-	0	Required by cooling tower	Weekly
Chlorine	-	0	-	0	Required by cooling tower	Weekly
Ion composition	-	0	Descaling needs	1,5 months	-	0

10



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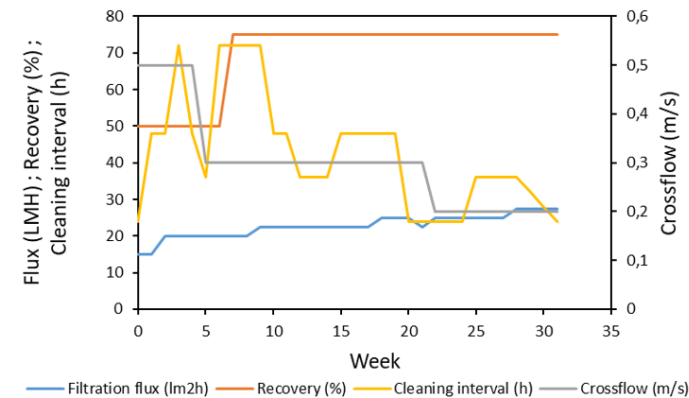
CS5: Operational procedures and methodologies

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Method for nanofiltration: change of operating conditions, by means of intensification of the filtration process.

Variable conditions:

- Flux
- Recovery
- Cleaning interval (frequency)
- Crossflow velocity



Method for reverse osmosis: stable conditions, although trying to maximize the recovery and the produced flow by means of pumping adjustment. Influence of dissolved COD on cleaning membranes. Simulation of RO through software.

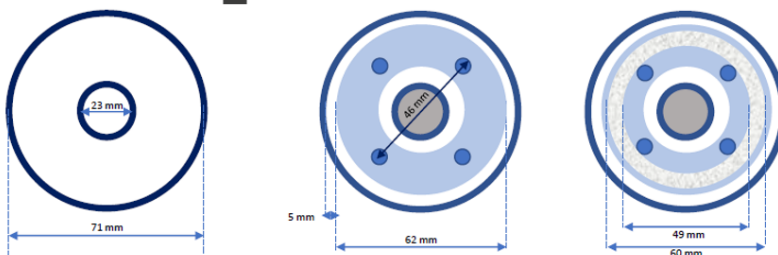




CS5: Development of AOP & UV test device

Subtask: 1.2.5 Reuse of brewery wastewater as process water

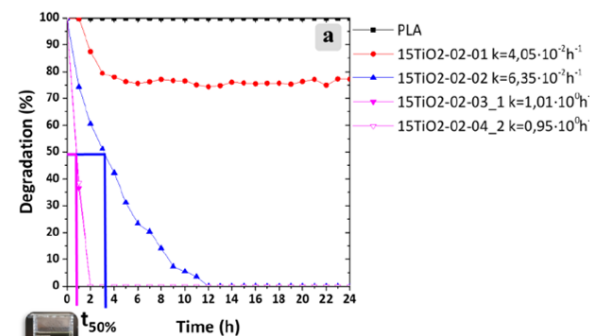
1. Photocatalytic reactor with support and first PLA prototypes adapted to the geometry of existing UV lamp.



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12

2. Design of ceramic filaments, printibles and sinterables, with high photocatalytic performance, adapted to the geometry of existing UV lamp.



Photodegradation of Methyl orange during 24h of sunlight exposition to rectangular TiO_2 membranes

3. Batch tests monitoring ofloxacin degradation with synthetic and real water.



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CS5: Pictures of AOP & UV test device

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Pump and structure with filaments



13



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CS5: Operational procedures and methodologies

Subtask: 1.2.5 Reuse of brewery wastewater as process water

Analytical plan

Determination of Ofloxacin (a quinolone antibiotic, $C_{18}H_{20}FN_3O_4$) concentration and UV absorbance in batch tests:

- Per triplicate
- Determination of the influence of support and support + membranes on results, compared with only UV activity.

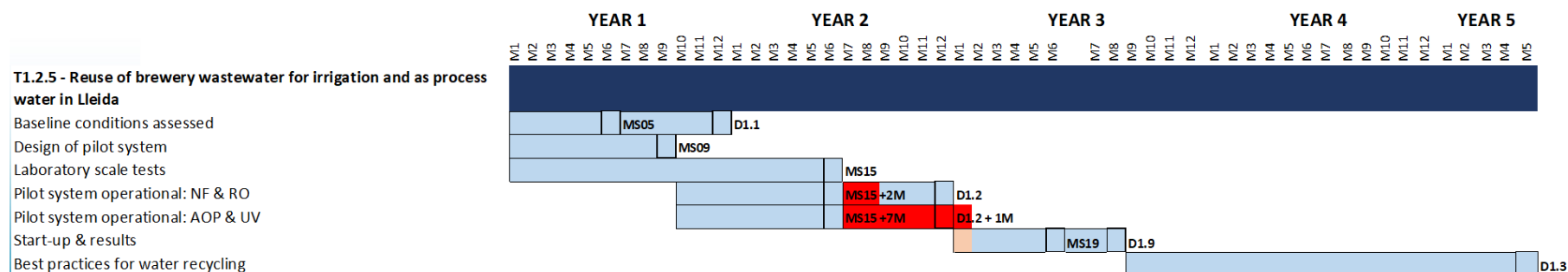
	Support	Membrane	Presence of TiO_2
Experiments 1&2	N	N	N
Experiments 3&4	Y	N	N
Experiments 5&6	Y	Y	N
Experiments 7&8	Y	Y	Y
Experiments 9&10	Y	Y	Y
Experiments 11&12	Y	Y	Y





CS5: Subtask 1.2.5 – Timeline

Subtask: 1.2.5 Reuse of brewery wastewater as process water



→ NF & RO are operational

→ AOP & UV are operational



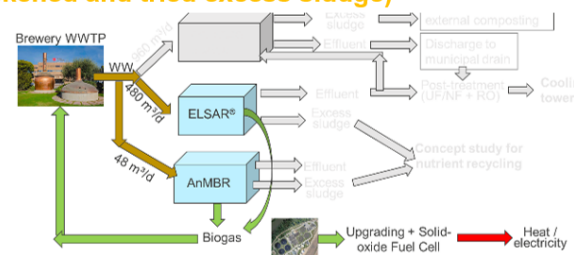


CS5: Subtask 1.3.2 Status/progress

Subtask: 1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell
Baseline technology: no energy production so far (only wastewater treatment with activated sludge process and subsequent composting of thickened and tried excess sludge)

Ultimate solutions to foster circular economy:

- Anaerobic membrane bioreactor (AnMBR),
- Electrostimulated anaerobic reactor (ELSAR),
- Solid oxide fuel cell (SOFC)



TRL: 7 → 9 (AnMBR); 5 → 7 (ELSAR); 7 → 9 (SOFC)

Capacity: 48 m³/d (AnMBR); 480 m³/d (full-scale ELSAR); 6 m³/d (pilot-scale ELSAR); 10 Nm³/d (SOFC)

Quantifiable targets: 20.000 m³ biogas/a (AnMBR); 200.000 m³ biogas/a (ELSAR); 4000-12.000 kWh_{el}/a (SOFC)
>100 % energy recovery

Status/progress:

- Running detailed design: online monitoring system.
- Operational: AnMBR, SOFC, pilot-scale ELSAR
- Building license received: full-scale ELSAR; construction expected to be completed in Sept. 2023





CS5: Pictures of the new technologies

Subtask: 1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell



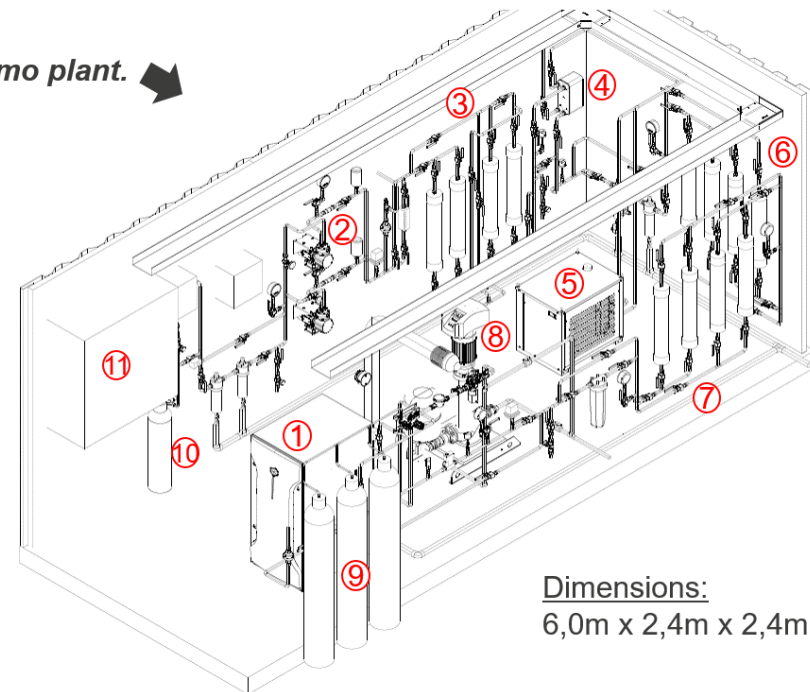
Solid-oxide fuel cell.

Supplier Solid Power; Model BlueGen BG-15.
Power output 0,5-1,5 kWe. Electrical efficiency > 57%.

Solid-oxide fuel cell demo plant.

Composed by:

1. Fuel Cell
2. Vacuum pumps
3. Desulphuration filters
4. Heat exchanger
5. Chiller
6. Dehumidification filters
7. Activated carbon filters
8. Pressure pump
9. Emergency biogas supply
10. Nitrogen gas
11. Electrical cabinet / PC



Dimensions:

6,0m x 2,4m x 2,4m





CS5: Pictures of the new technologies

Subtask: **1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell**



SOFC pilot plant installed in WWTP Lleida

18



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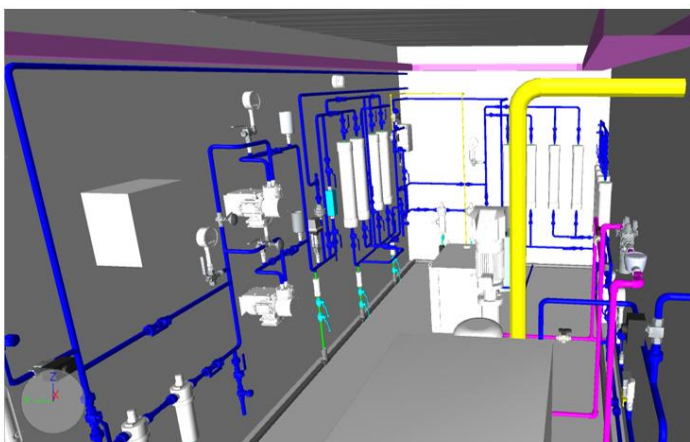


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CS5: Pictures of the new technologies

Subtask: **1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell**



What was intended to do: 3D view of the SOFC pilot plant in engineering project



What has been done: real picture of the SOFC pilot plant (taken April 2022)

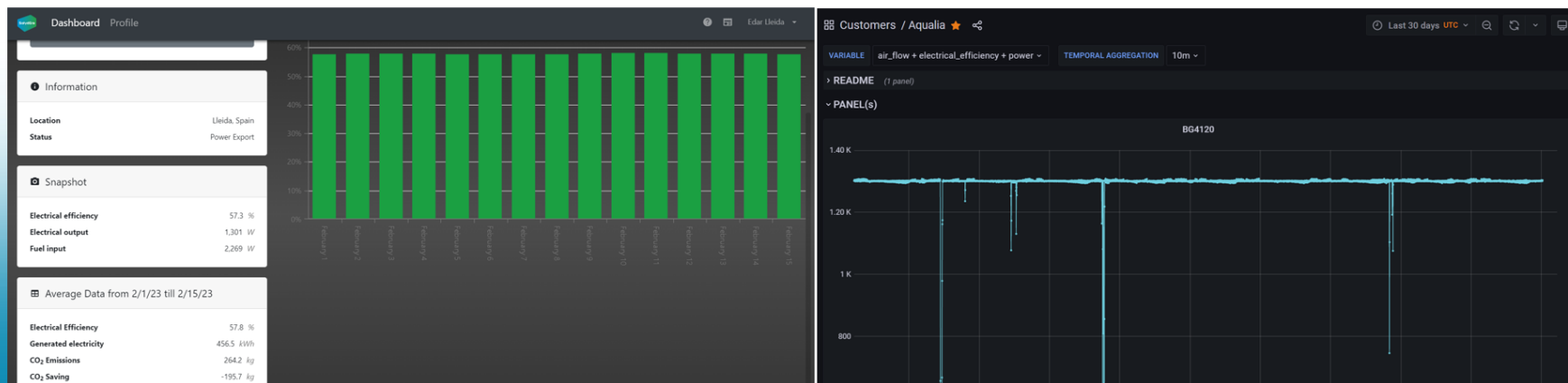
- ➡ In November 2022:
1. Final integration of fuel cell
 2. Hot start-up (biogas) → Operation





CS5: Pictures of the new technologies

Subtask: 1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell



→ First 2 months of operation show a constant and expected output's behaviour:

→ Constant electrical efficiency between 57,5- 57,9%

→ Produced power 1,3 kW

→ Slight adjustments for optimization are to be done: automatization, measuring, etc.



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CS5: First results

Subtask: 1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell

On 01.12, the first characterization of the biogas in WWTP Lleida was carried out before and after being subjected to the pre-treatment (iron salts + zeolites + activated carbon), in order to verify the performance of the proposed pre-treatment. The main results are:

- Moisture removal from 10,800 ppm to 5,000 ppm.
- No presence of siloxanes ($<0.05 \text{ mg / Nm}^3$) neither in input nor in pre-treated biogas.
- Total removal of H_2S (80.3 mg/Nm^3 inlet)
- Presence of ammonia, with slight concentration increase (from 8.8 to 11.4 mg / Nm^3)
- No presence of VOC in the input ($<0.02 \text{ mg / Nm}^3$), but surprisingly a small concentration in pre-treated biogas (1 mg / Nm^3).

Further analytical determination is going to be done in 2023.

→ **Pre-treatment of biogas before entering the SOFC is validated.**

21



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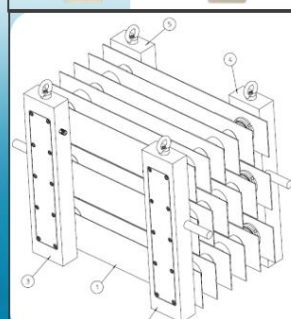
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CS5: Pictures of the new technologies

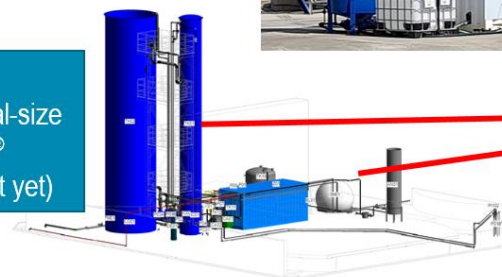


ELSAR® Prototype: Lab-scale (5L) and pilot scale (1 m³).



Pilot-scale electrode installed end of 2022

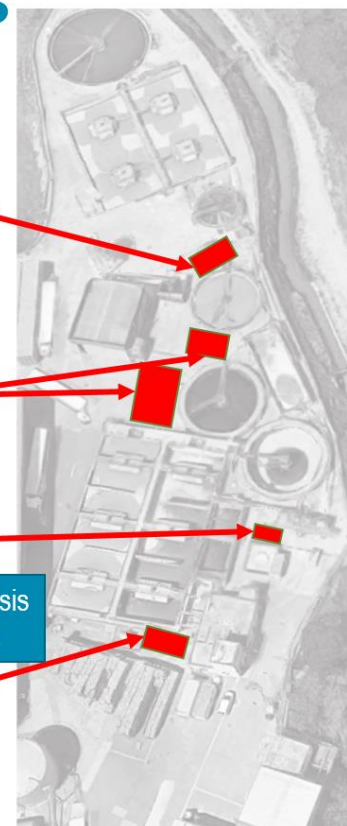
Future industrial-size ELSAR® (not built yet)



Nanofiltration (not any more)



Reverse osmosis (not any more)



CS5 plants installed (or to be installed) in the brewery Mahou San Miguel



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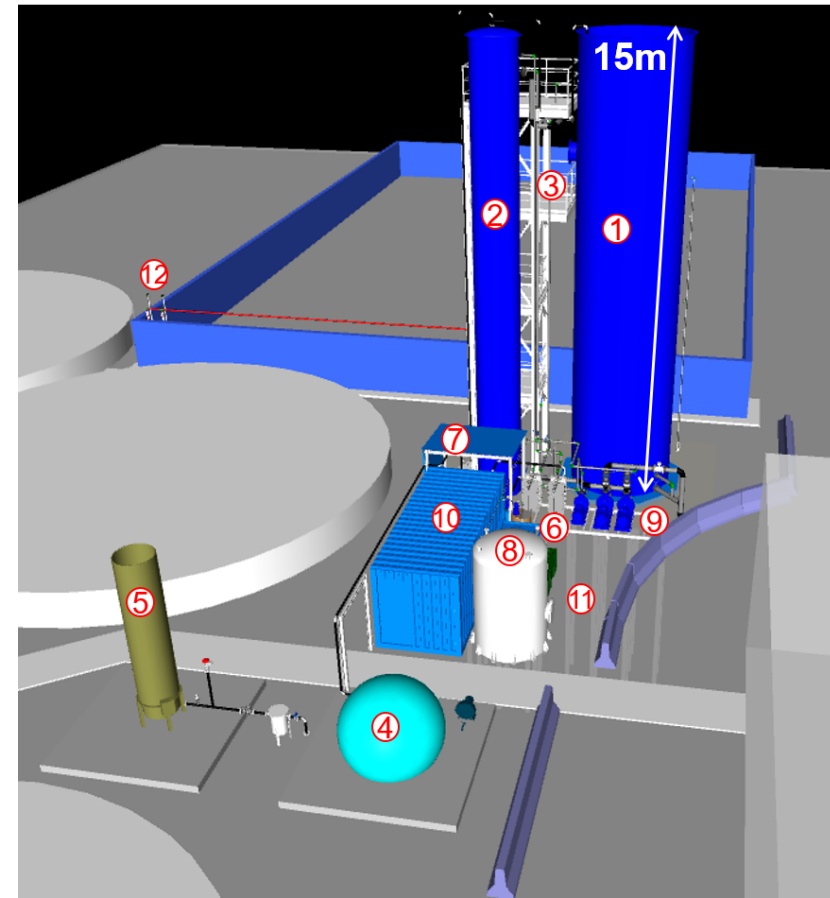
CS5: Pictures of the new technologies

Subtask: **Electrostimulated anaerobic reactor (ELSAR®)**

- **Capacity**
 - Input Brewery Wastewater
 - Flow 20 m³/h, OLR 2 Tn COD/d
- **Reactor features**
 - Total Volume Reactor 140m³
 - Ø 3,5m; Water height 15m
 - Mesophilic range (30 - 37°C)
- **Expected results**
 - 90% COD removal
 - 31 Nm³ biogas/h
 - Energy surplus

Composed by:

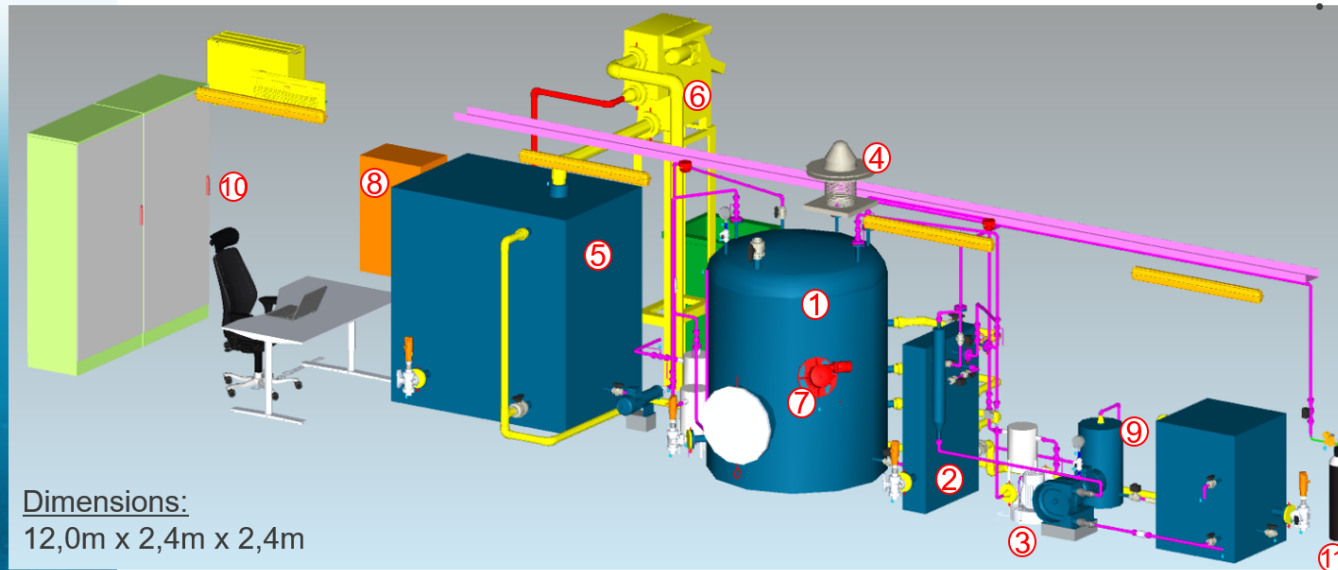
1. ELSAR reactor
2. Buffer reactor
3. Stairs structure
4. Gazometer
5. Flare
6. Heat exchangers
7. Chiller
8. Chemical storage
9. Pumping station
10. Office, lab, store
11. Foundation
12. Feeding pumps





CS5: Picture of the AnMBR

Subtask: **Anaerobic Membrane Bio Reactor (AnMBR)**



Dimensions:
12,0m x 2,4m x 2,4m

- **Capacity**
 - *Input Industrial Wastewater*
 - *Flow 2 m³/h*
 - *OLR 200 kg COD/d*
- **Reactor features**
 - *Total Volume Reactor 40m³*
 - *Mesophilic range (30 - 37°C)*
- **Expected results**
 - *95% COD removal*
 - *3,5 Nm³ biogas/h*

Composed by:

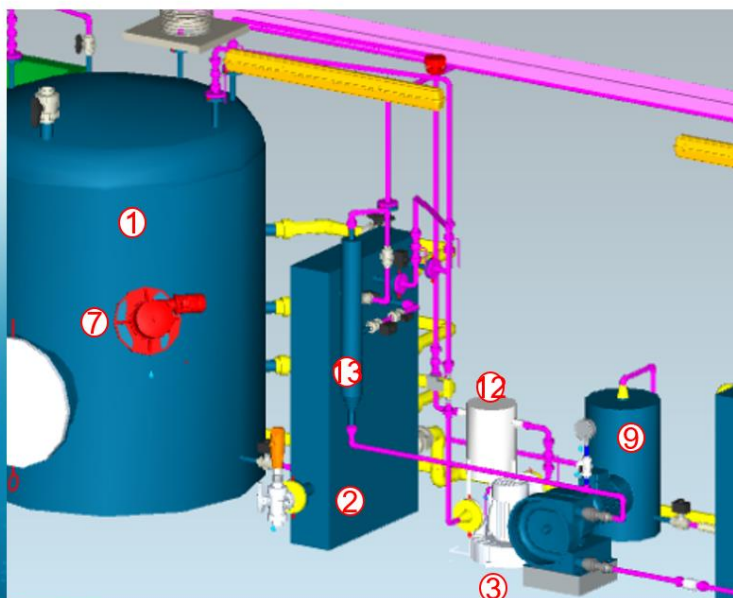
1. Biological reactor
2. Membranes
3. Blower and recirculation pumps
4. Ventilator
5. Buffer tank
6. Screen
7. Stirrer
8. Electrical cabinet
9. Backwash and permeate tanks
10. Office
11. Inert gas



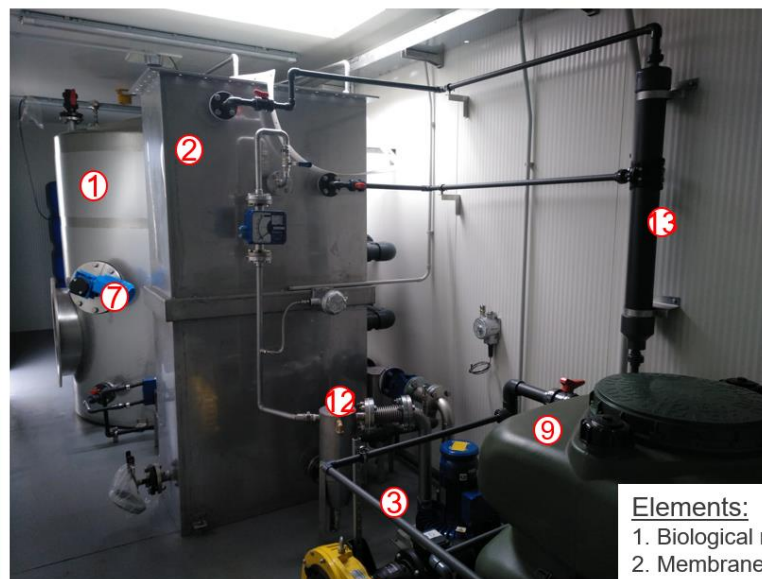


CS5: Pictures of the AnMBR

Subtask: **Anaerobic Membrane Bio Reactor (AnMBR)**



What was intended to do: 3D view of the AnMBR pilot plant in engineering project



What has been done: real picture of the AnMBR pilot plant (taken April 2022)

Elements:

- 1. Biological reactor
- 2. Membranes
- 3. Blower + recirculation pump
- 7. Stirrer
- 9. Backwash / permeate tanks
- 12. Condensates pot
- 13. Degassing unit





CS5: Operational procedures and methodologies

Subtask: **1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell**

SOFC

- **Monitoring of:**
 - Monthly analytical determination of biogas components (before entering the SOFC).
 - Online measuring of pressure, temperature and moisture before entering the SOFC.
 - Register of biogas consumption, produced energy, electrical energy consumption and water consumption.
- **Support:** Training and online support of the SOFC will be provided by the supplier during the first operation year.
- **Security measures:**
 - Excess air ventilation
 - 2 units of lower explosive limit (LEL) detector for CH₄
 - Flame arresters

26



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ELSAR® and AnMBR

- **Monitoring of:**
 - Weekly analytical determination of produced biogas components and of treated wastewater.
 - Online measuring of fouling-linked parameters (only for AnMBR) as well as several operational parameters.
 - Operation without and with the electrochemical system, at different voltage (only for ELSAR®).
 - Register of chemical consumption, produced energy and electrical energy consumption.
- **Security measures:**
 - Excess air ventilation (only for AnMBR)
 - Lower explosive limit (LEL) detectors for CH₄ (only for AnMBR)
 - Flame arresters





CS5: Operational procedures and methodologies

Subtask: **ElectroStimulated Anaerobic Reactor (ELSAR®) and Anaerobic Membrane Bio Reactor (AnMBR)**

Due to postponement of building permission obtention (12 months waiting time, instead of max. 5 months), the following measures have been taken:

- Extension of lab- and pilot-scale experiments to accelerate start-up and optimization phases of full-scale ELSAR®



*Picture of the pilot plant in WWTP Mahou SM.
Average results in February 2023 (tests without ElectroQ).*

Conditions		Results	
Parameter	Value	Parameter	Value
Temperature	36,3°C	TCOD removal	89,9%
pH	8,93	TSS removal	43,8%
ORP	-73mV	Methane productivity	0,38 m ³ CH ₄ /kg removed COD
OLR	3,6 kg COD/m ³ /d		





CS5: Operational procedures and methodologies

Subtask: **ElectroStimulated Anaerobic Reactor (ELSAR®) and Anaerobic Membrane Bio Reactor (AnMBR)**

The inoculation of anaerobic sludge was made in Dec. 2022. So far it has been fed in batch mode, feeding small volumes of municipal wastewater, without (ultra)filtering. After consolidating the biomass, starting March 2023 the filtration will be activated.



From left to right:

- **Manhole for wastewater suction**
- **Level of digester**
- **Gas counter**
- **Control panel**

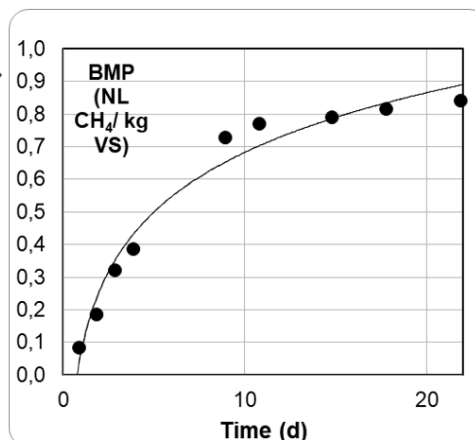




CS5: Laboratory results

Subtask: **Electrostimulated anaerobic reactor (ELSAR®) and Anaerobic Membrane Bio Reactor (AnMBR)**

- Exhaustive brewery wastewater characterization (1 month long)
- Biochemical methane potential (BMP) tests showing adequate anaerobic biodegradability. A potential of 0,31 Nm³ CH₄/ removed kg COD was found. This result is consistent with other sources.
- Preliminary geotechnical study & basic design projects shows no technical limitations for proposed solutions (but certain need for foundation & civil works)



PARAMETER	AVERAGE ± STANDARD DEVIATION	UNITS
COD (stirred sample)	5586±1732	mg/L
COD (settled sample)	4674±1765	mg/L
NH ₄	3±3	mg/L
NO ₃	2±1	mg/L
Total N	64±23	mg/L
Total P	17±4	mg/L
Sulphates	158±32	mg/L
Sulphur	<1	mg/L
Conductivity	2551±627	µS/cm
Total alkalinity	19,3±6,6	meq/L
Partial alkalinity	8,8±4,4	meq/L
Intermediate alkalinity	12,9±3	meq/L
Volatile fatty acids	15±3,6	mg Ac/L
pH	6,67±0,96	-log[H ⁺]
Total suspended solids	199±99	mg/L
Volatile suspended solids	124±51	mg/L
% SSV	0,67±0,16	%
Settled solids	40±30	mg/L





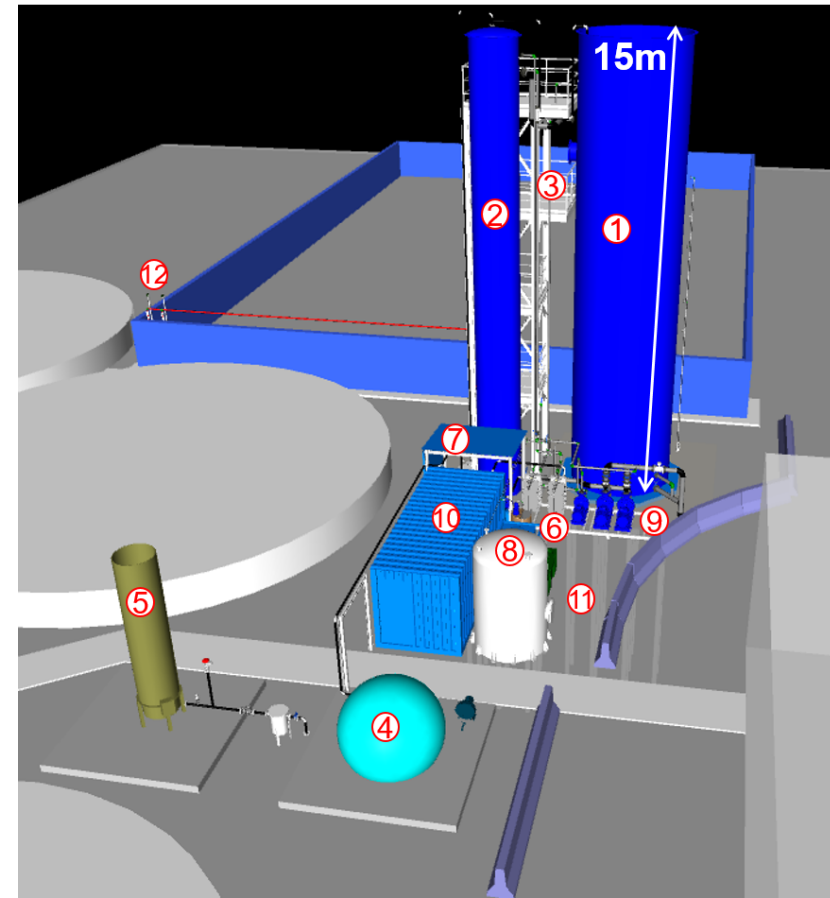
CS5: full-scale ELSAR

Subtask: **Electrostimulated anaerobic reactor (ELSAR®)**

- **Capacity**
 - *Input Brewery Wastewater*
 - *Flow 20 m³/h, OLR 2 Tn COD/d*
- **Reactor features**
 - *Total Volume Reactor 140m³*
 - *Ø 3,5m; Water height 15m*
 - *Mesophilic range (30 - 37°C)*
- **Expected results**
 - *90% COD removal*
 - *31 Nm³ biogas/h*
 - *Energy surplus*

Composed by:

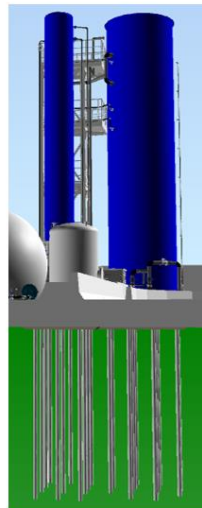
1. ELSAR reactor
2. Buffer reactor
3. Stairs structure
4. Gazometer
5. Flare
6. Heat exchangers
7. Chiller
8. Chemical storage
9. Pumping station
10. Office, lab, store
11. Foundation
12. Feeding pumps





CS5: Pictures of full-scale ELSAR construction

Subtask: **Electrostimulated anaerobic reactor (ELSAR®)**



Drawing of the micropiles below the ELSAR and the buffer tank (left). Building of micropiles (right).

- Due to the height of the reactor, there is a need of deep civil works (micropiles)





CS5: Pictures of full-scale ELSAR construction

Subtask: **Electrostimulated anaerobic reactor (ELSAR®)**



Pumps before mechanical assembly.



The retaining wall for emergency NaOH leakages.



Flare being installed.

32



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CS5: Full-scale ELSAR



Concrete platforms for torch, gasometer and lamellar clarifier

- Civil works expected to finish in June 2023
- New additional structures were required: lamellar clarifier, retaining wall for emergency NaOH leakages
- Most of the electromechanical components are on site
- Mechanical and electrical assembling currently being done

33



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Buffer tank (left) and reactor (right).
On the bottom left the retaining wall for emergency NaOH leakages can be seen



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CS5: Subtask 1.3.2 – Timeline for AnMBR & SOFC

Subtask: 1.3.2 Anaerobic pretreatment of brewery wastewater and electricity production via solid-oxide fuel cell

T1.3.2 - Anaerobic treatment of brewery and food industry wastewater as well as biowaste to recover biogas in Lleida

Baseline conditions assessed

Design of pilot system

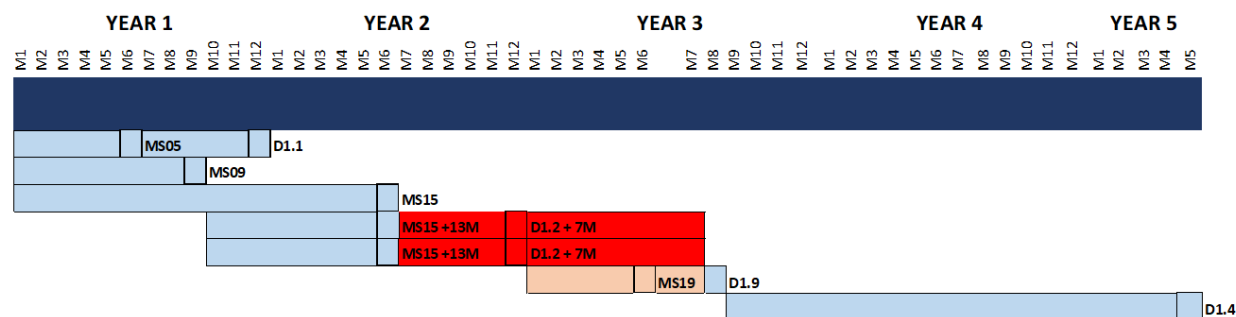
Laboratory tests & investigations

AnMBR operational

SOFC operational

Start-up & results

Best practices for energy recovery



→ AnMBR & SOFC are operational since Dec. 2022



Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity





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CS5: Subtask 1.4.4 Status/progress

Subtask: 1.4.4 Recovery of nutrients from brewery digestates

Baseline technology: composting of thickened and tried excess sludge

Ultimate solution to foster circular economy:

1. STRUVITE / VIVIANITE

Feasibility of integration of Aqualia technologies and previous experiences

2. HYDROCHAR

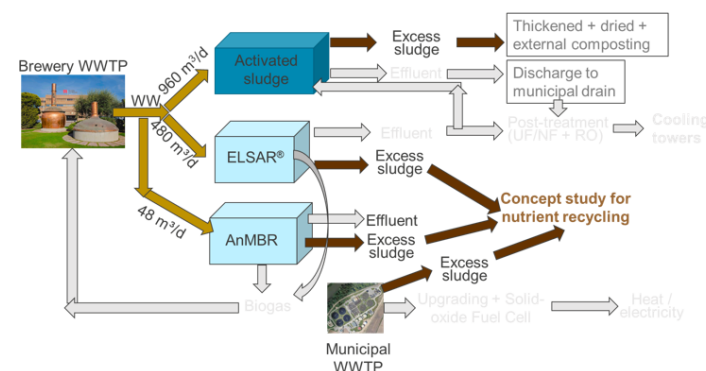
Sludge and other potential solids: spent grain + yeast, *tbd*;
Feasibility of integration and techno-economical comparison.
Special focus on solar-based HTC technologies

TRL: 5 → 7 (concept study: material recovery)

Capacity: P-recovery: 6 t phosphorous/a; **Hydrochar:** 600 t (brewery)/a & 1600 t (WWTP)/a

Quantifiable target: 6 t phosphorus/a; 6% P recovery; 600 t hydrochar/a

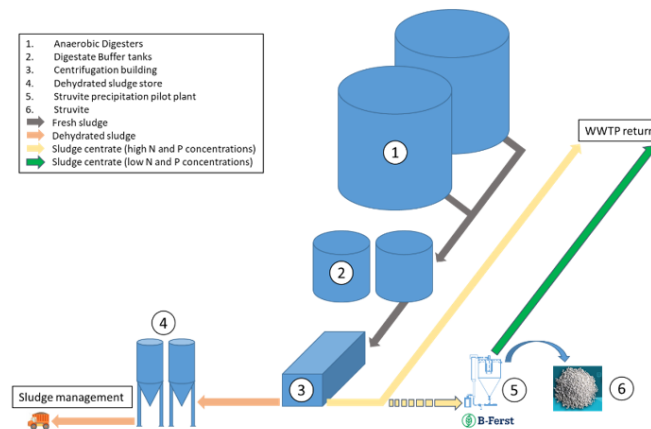
Status/progress: Feasibility report under progress.





CS5: Concept study incl. solar pilot plant

Subtask: 1.4.4 Recovery of nutrients from brewery digestates

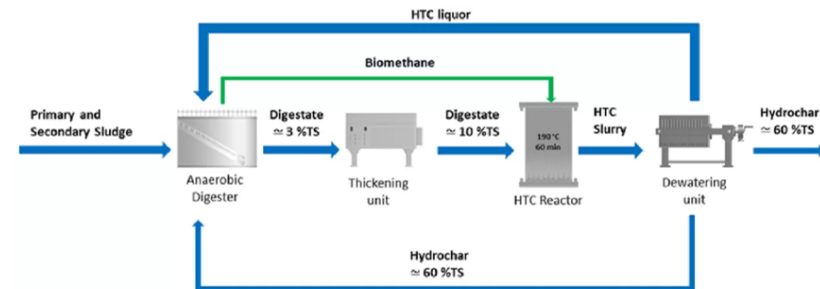


1. STRUVITE / VIVIANITE

- Potential of 6 T P/a in urban WWTP
- Feasibility of integration of Aqualia technologies and previous experiences



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



2. HYDROCHAR

- Potential sludge 600 T/a (brewery) & 1600 T/a (urban WWTP) (dry basis)
- Potential 1 GWh/a of effective solar energy used for HTC.
- Other potential solids: spent grain + yeast, *tbd*
- Feasibility of integration and techno-economical comparison

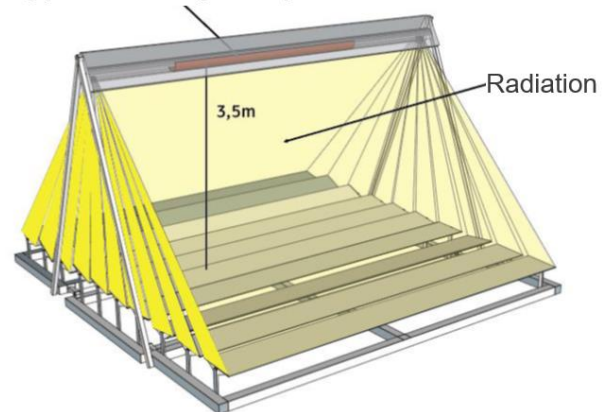




CS5: Pictures of the solar pilot plant

Subtask: 1.4.4 Recovery of nutrients from brewery digestates

Testing plate for sludge samples



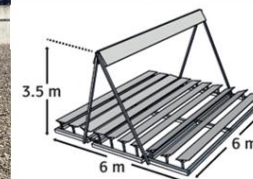
What was intended to do: 3D view of the solar pilot plant in engineering project



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What has been done: real picture of the solar pilot plant (taken April 2022)



Supplied power (based on max. typical climate data)	14,5 kWt
Net mirror surface	26,4 m ²
Footprint	36 m ²
Expected lifespan	20 years
Monitoring of energetic production & climatic data	Yes
Self-orientation of mirrors	Yes
Remote visualization	Yes





CS5: Operational procedures and methodologies

Subtask: 1.4.4 Recovery of nutrients from brewery digestates

Concentrated solar pilot plant for sludge treatment

- **Monitoring of:**
 - Temperature
 - Moisture (for drying evaluation)
 - Volatile matter (for hydrolysis evaluation & carbonization)
 - *E. Coli*, *Samonella ssp.*, *Clostridium perfringens* (for disinfection evaluation), contrasting with EC draft.
- **Evaluation of results:**
 - Monitored variables at different set temperatures will be contrasted with models
- **Mode:**
 - Batch tests → proof of concept
 - Development & test of a continuous system

39



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Example of screen of the remote visualization

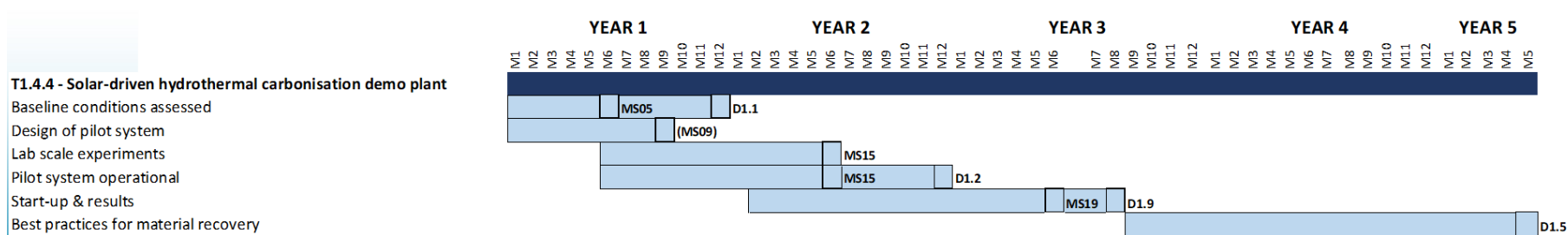
	<i>E. coli</i>	<i>Salmonella</i>	<i>Clostridium</i>
	CFU/g DM	Presence in 50g	(CFU/g DM)
Draft EU 86/278/CEE	< 10 ³	NO	< 3·10 ³





CS5: Subtask 1.4.4 – in time

Subtask: 1.4.4 Recovery of nutrients from brewery digestates



→ Solar pilot plant is operational since May 2022

Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity

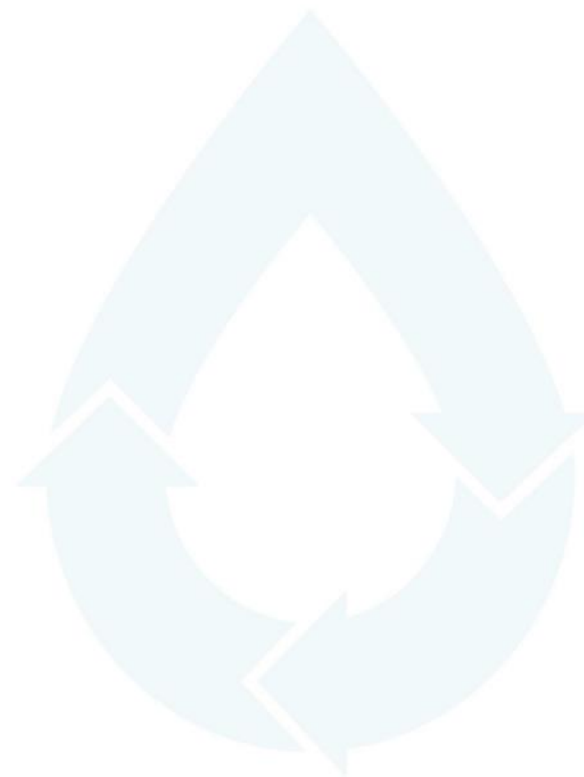




WATER SMART INDUSTRIAL SYMBIOSIS

CS5 Contact

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2.6. CS6: Karmiel & Shafdan

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
6	1.3.3	AAT Karmiel		100%	100%	May 22
	1.3.4	AAT + membrane filtration incl. PAC Shafdan	90%	100%	100%	Aug 22
	1.4.5	Recovery polyphenols (pilot system: adsorption column)	100%	100%	100%	Dec. 22



D1.2 Operational demo cases CS6 Shafdan & Karmiel

GSR, GtG, MEK, AGROBICS



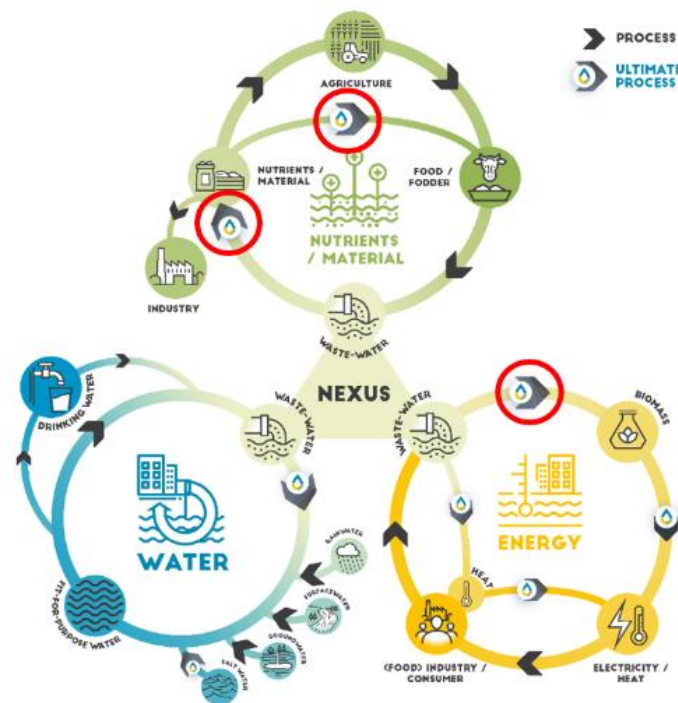


CS6: Karmiel and Shafdan

Lead partner:



Other partners:



2



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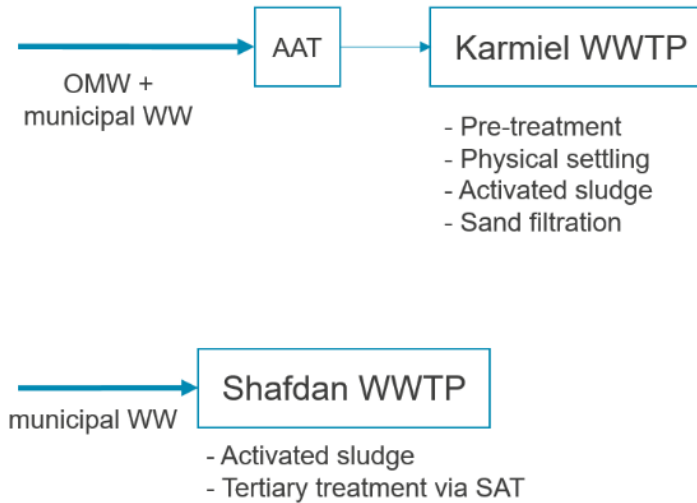


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CS6: Situation before Ultimate

Existing demo scale



3



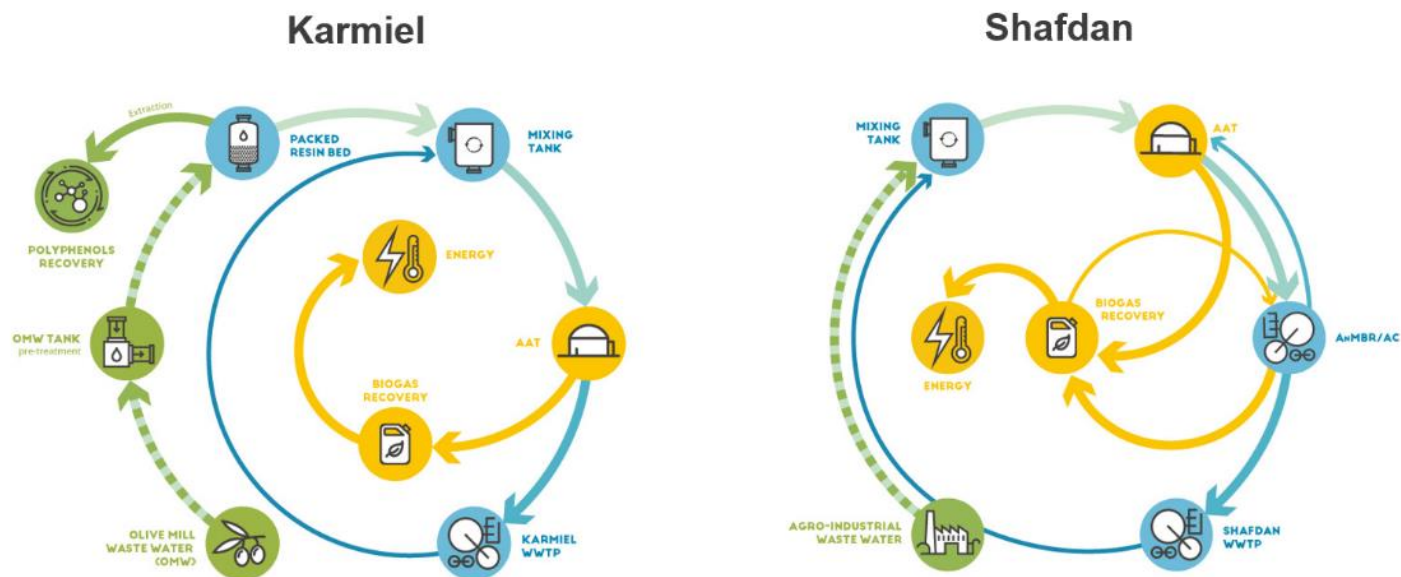
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CS6: Objectives of the Ultimate solutions



4



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CS6: Subtask 1.3.3 Status/progress - Karmiel

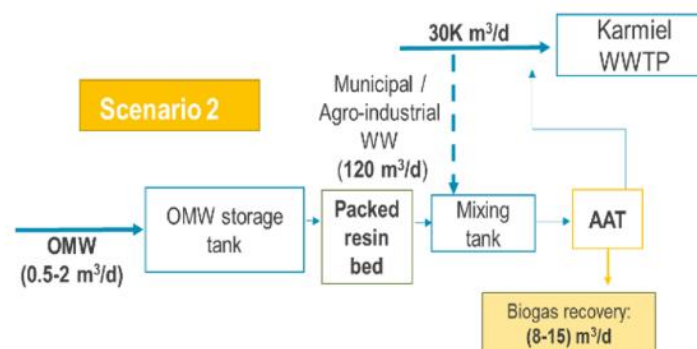
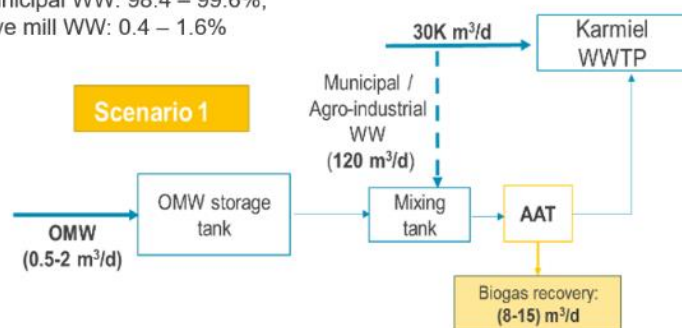
Subtask: 1.3.3 Biogas production from anaerobic pre-treatment of municipal and/or industrial wastewater in Karmiel

Baseline technology: Existing AAT demonstration plant

Ultimate solution to foster circular economy: Advanced Anaerobic Technology (AAT) for biogas production

Capacity: 120 m³/d TRL: 5 → 8

municipal WW: 98.4 – 99.6%;
olive mill WW: 0.4 – 1.6%



Quantifiable targets: 8-15 m³ biogas/d; 20-25% reduction of energy demand; 25% energy recovery

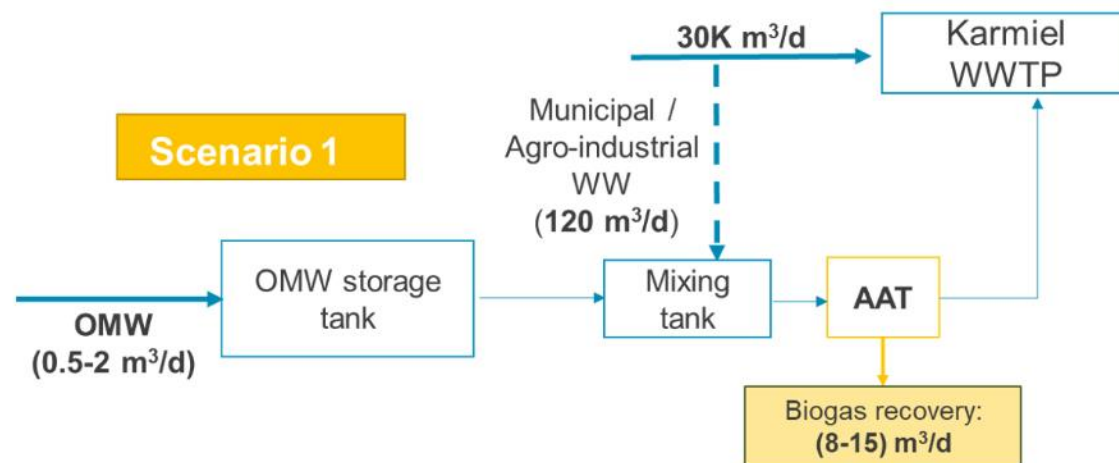
Status/progress:

- detailed design completed
- constructed and operational



CS6: Current operational procedures and methodologies - Karmiel

Subtask: 1.3.3 Biogas production from anaerobic pre-treatment of municipal and/or industrial wastewater in Karmiel



6



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AAT ... Advanced anaerobic treatment
OMW... Olive mill wastewater
WWTP... Wastewater treatment plant





CS6: Picture of the high rate anaerobic reactor (AAT)

Subtask: 1.3.3 Biogas production from anaerobic pre-treatment of municipal and/or industrial wastewater in Karmiel



7



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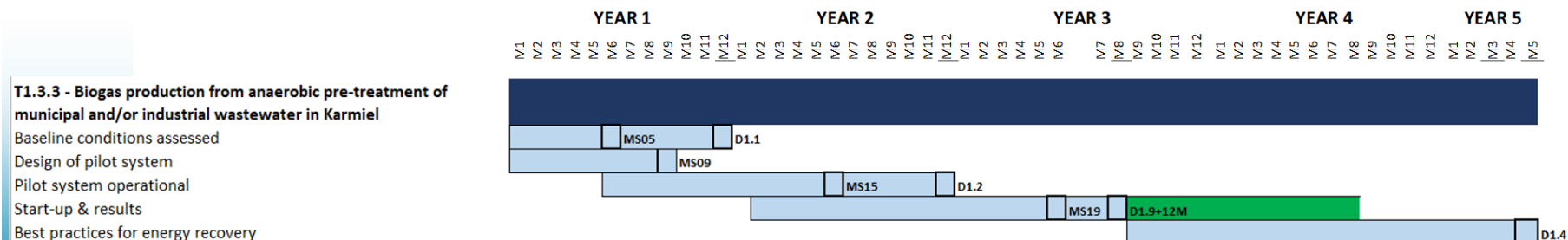


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CS6: Task 1.3.3 is in time - Karmiel

Subtask: **1.3.3 Biogas production from anaerobic pre-treatment of municipal and/or industrial wastewater in Karmiel**



→ Immobilized anaerobic high-rate reactor (AAT) is operational



Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity





CS6: Subtask 1.3.4 Status/progress - Shafdan

Subtask: 1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan

Baseline technology: Biogas production via existing anaerobic digestion (AD)

Ultimate solution to foster circular economy: AAT with AC to prevent biomass inhibition

TRL: 5 → 7

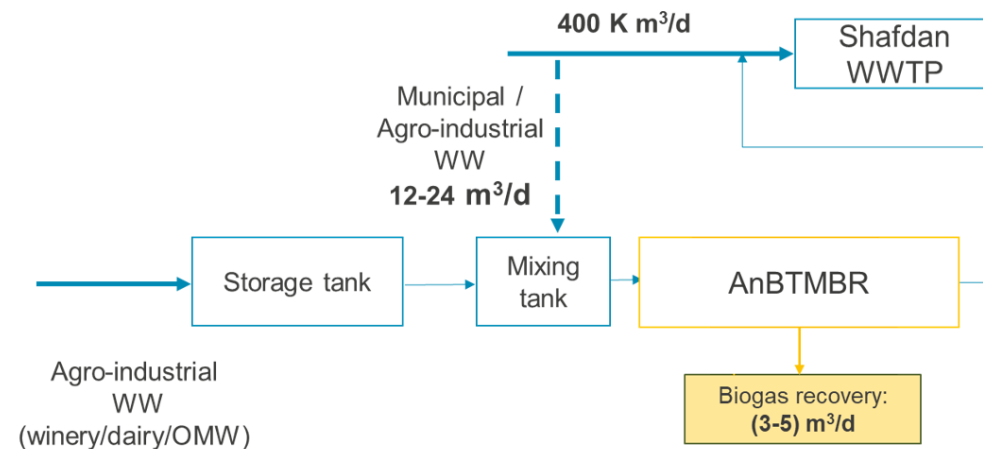
Capacity: 12-24 m³/d

Quantifiable targets:

- 3-5 m³ biogas/d;
- 20-25% reduction of energy demand;
- 25% energy recovery

Status/progress:

- Detailed design completed
- Constructed and operational



AnBTMBR ... Anaerobic biofilm treatment membrane bioreactor

OMW... Olive mill wastewater

WWTP..... Wastewater treatment plant

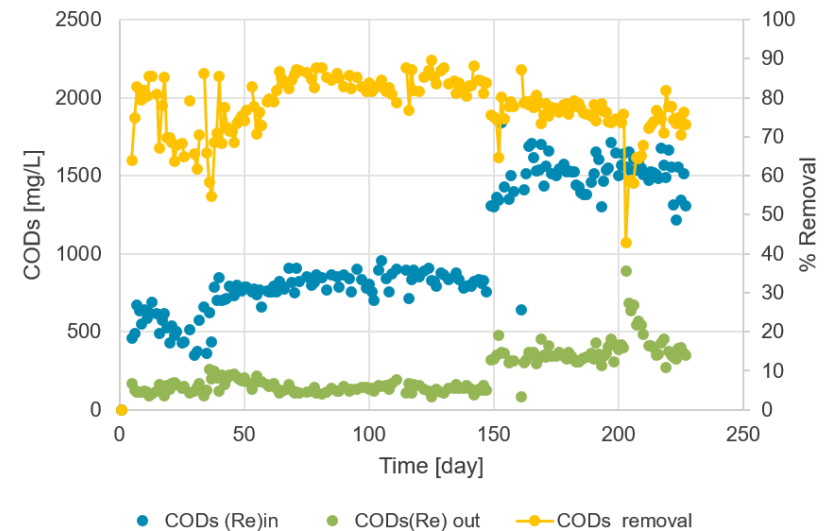




CS6: Results of the laboratory experiments

Subtask: 1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan

The lab-scale: The start-up of the system has been. Below you can see the picture of the lab-scale system with results of the soluble COD removal by the lab scale AAT.

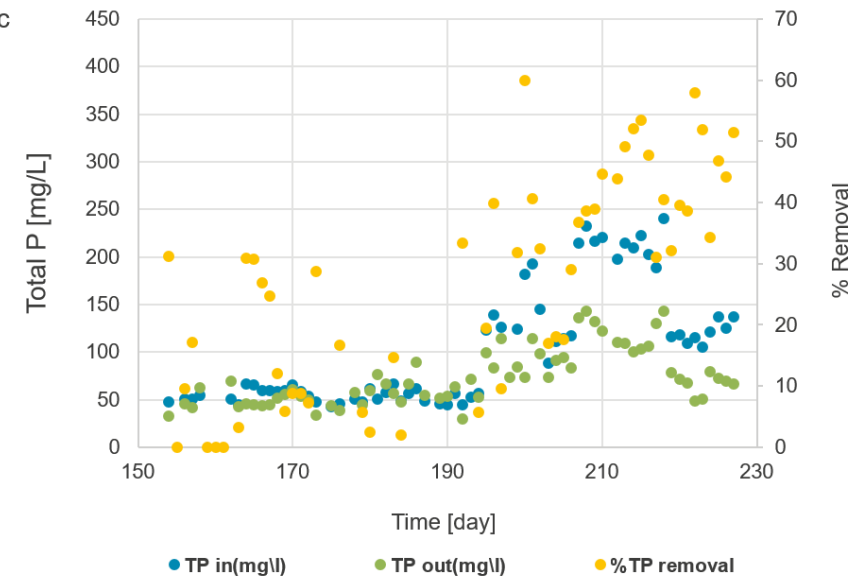




CS6: Results of the laboratory experiments

Subtask: 1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan

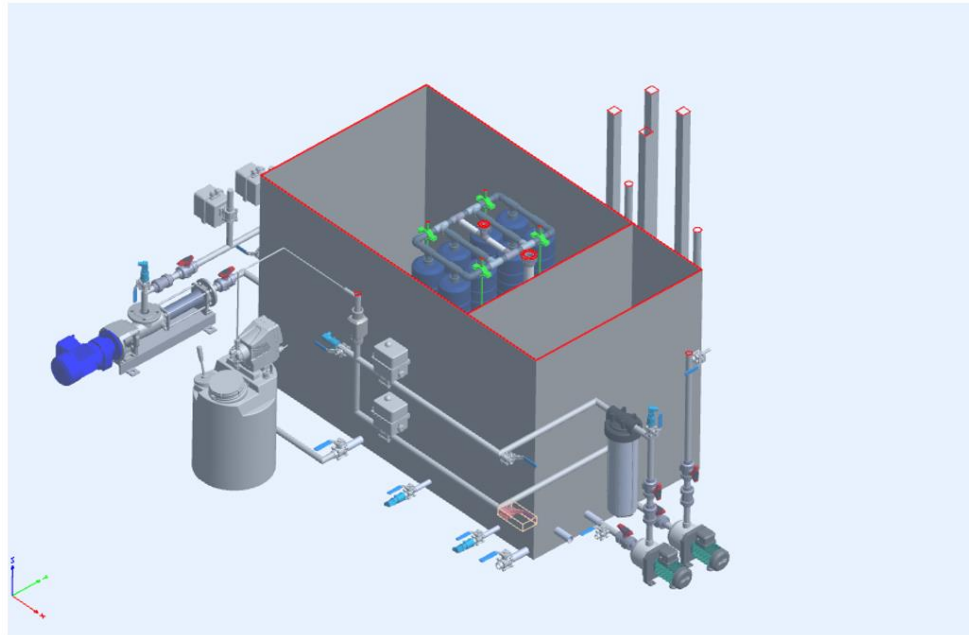
The lab-scale: The start-up of the system has been. Below you can see the picture of the lab-scale system with results of the total phenolic content removal by the lab scale AAT.





CS6: Pictures of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



12



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CS6: Pictures of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



13



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CS6: Pictures of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



14



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CS6 Video: construction of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**

*This video is accessible via the indicated link below this presentation on the **ULTIMATE** webpage.*





CS6: Pictures of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



16



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CS6: Pictures of the anaerobic biofilm treatment membrane bioreactor system

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



17



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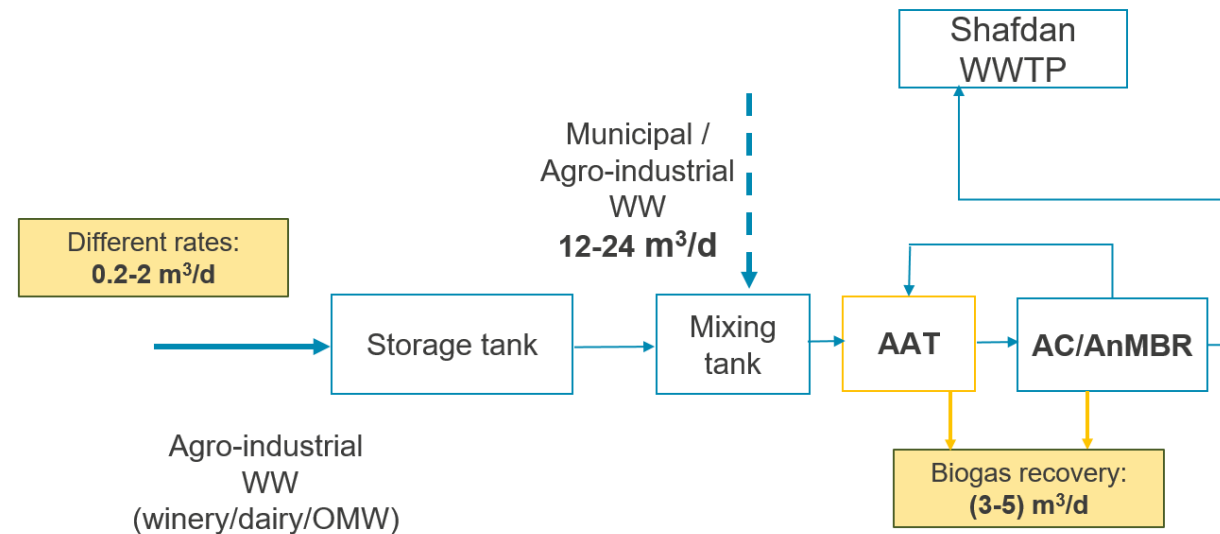


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CS6: Operational procedures and methodologies Shafdan

Subtask: 1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan





Start-up process of the Shafdan system

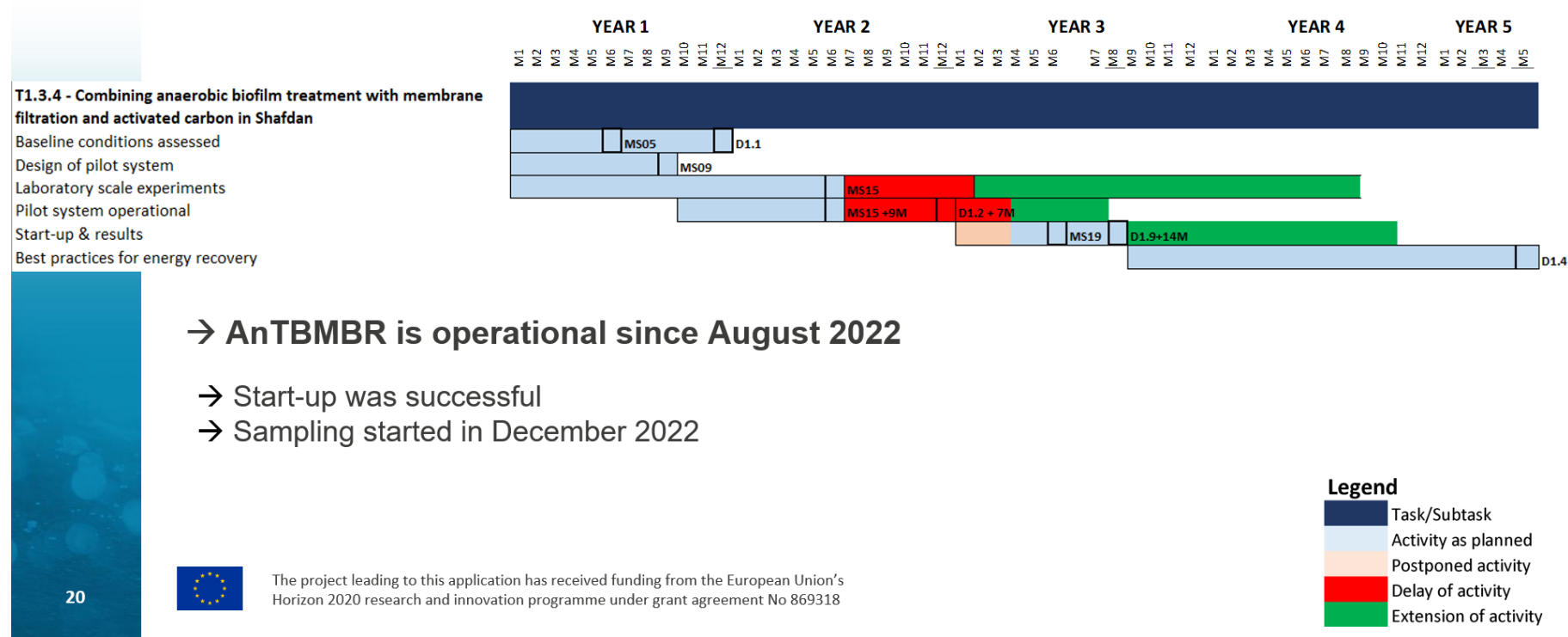
- After connecting the system with the inlet of wastewater (after filtration), the AAT system was started on August 2022 in order to reach a steady state of the anerobic unit (fixed foam-based AAT system) before connecting it to the filtration (Membrane unit). From August-October, 2022
- The filtration system (membrane) was mechanically and electrically commissioned, a deep check of the sensors and valve was done. Many issues related to the sensors, electrical panel and control system took about a month to be solved with the support of SFC staff (until end of September 2022)
- The membrane unites were checked with tap water with the support of the engineer of SFC Umwelttechnik GmbH (provider of the AnMBR system), where the connections and membrane.
- After then, the membrane system was connected to the outlet of the AAT system (on November 7th, 2022) and first complete run was started with 0.5 m³/h. From this time until November 16th, an intensive training on the automatic operation/monitoring was given by the SFC staff to the technical team of GS/Agrobic/Mekorot.
- On Nov. 21st 2022, we started the sampling camping according to the sampling plan.





CS6: Task 1.3.4 - Timeline - Shafdan

Subtask: **1.3.4 Combining anaerobic biofilm treatment with membrane filtration and activated carbon in Shafdan**



→ AnTBMBR is operational since August 2022

→ Start-up was successful

→ Sampling started in December 2022





CS6: Subtask 1.4.5 status/progress

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel

Baseline technology: No material recovery so far

Ultimate solution to foster circular economy:

Adsorption column with packed resin bed and extraction with pressurized hot water

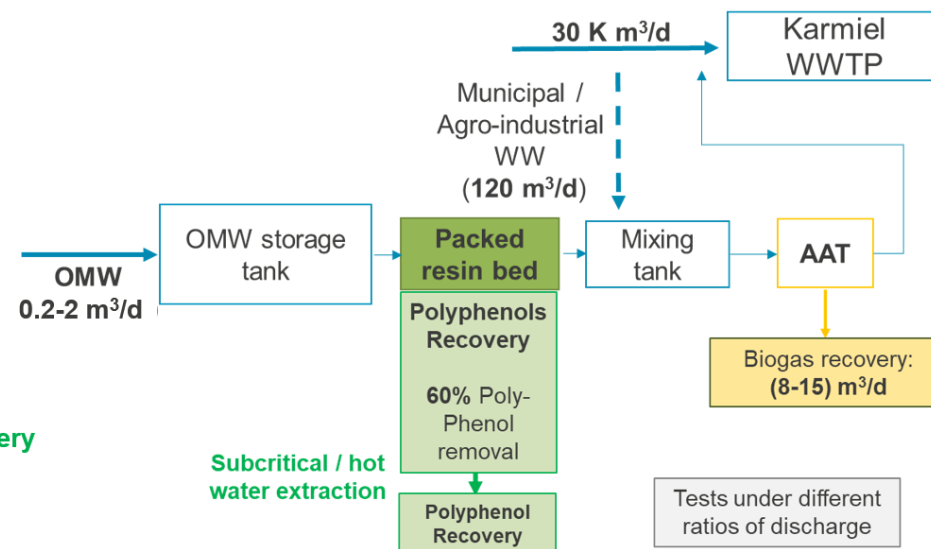
TRL: 5 → 7

Capacity: 0.2-2 m³/d

Quantifiable targets: > 40% Polyphenols recovery

Status/progress:

- Pilot plant operational
- Lab scale experiments completed





CS6: Pictures of the new technologies

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel



Static adsorption

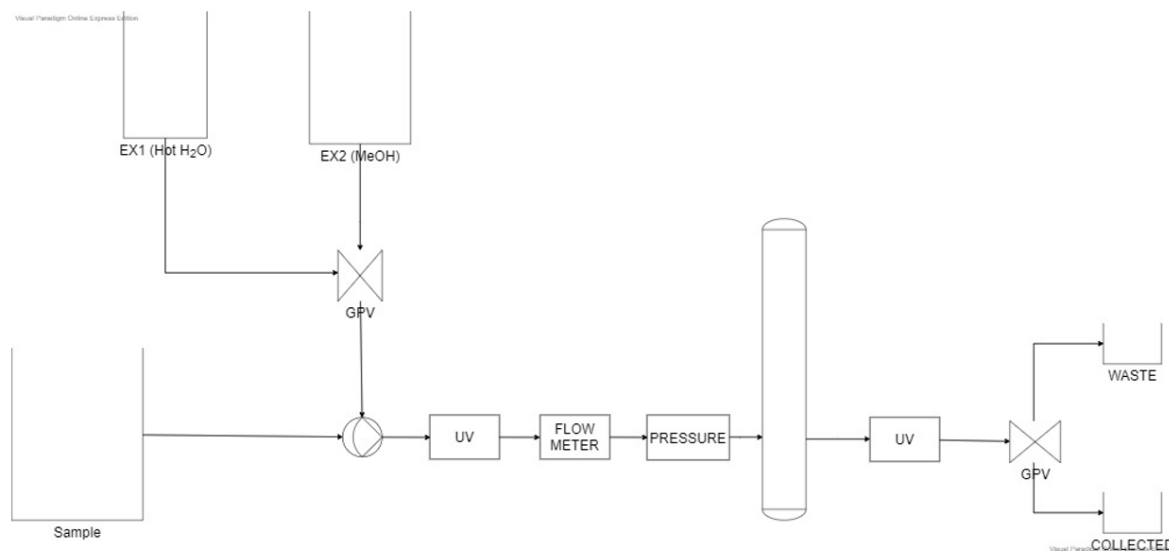
Lab scale – Dynamic adsorption





CS6: Operational procedures and methodologies

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel



23



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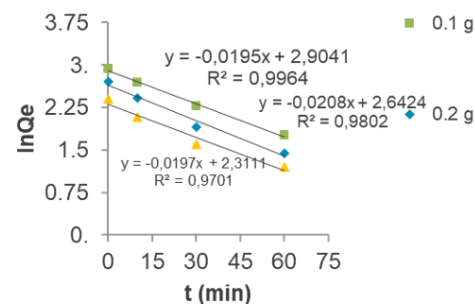
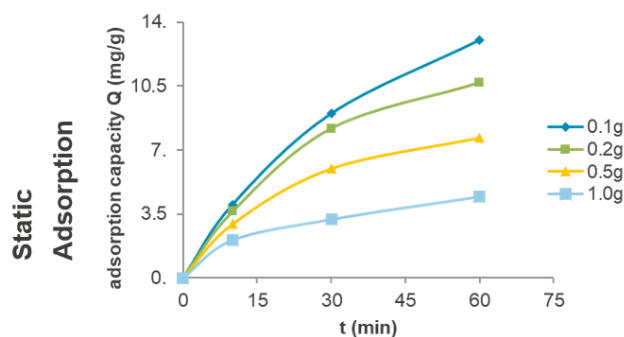


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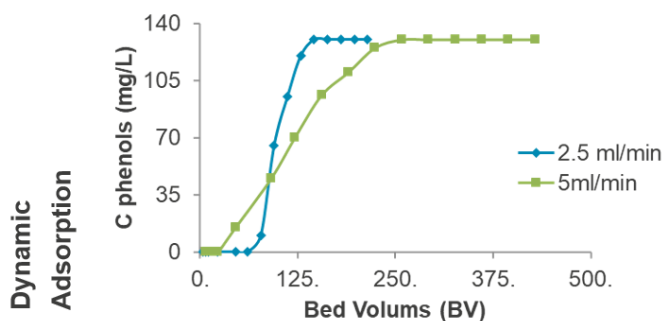


CS6: Results of the laboratory experiments

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel



Maximum adsorptive capacity (Q): 23 g of polyphenols per kg of resin for the FPX 66 resin



The breakthrough curves showed that 1.7 m³ of wastewater can be treated per kg of resin per 10 cycles

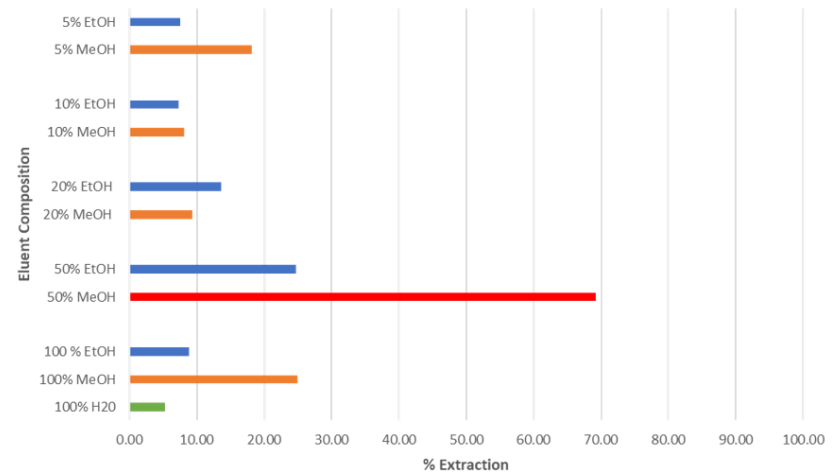




CS6: Laboratory results

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel

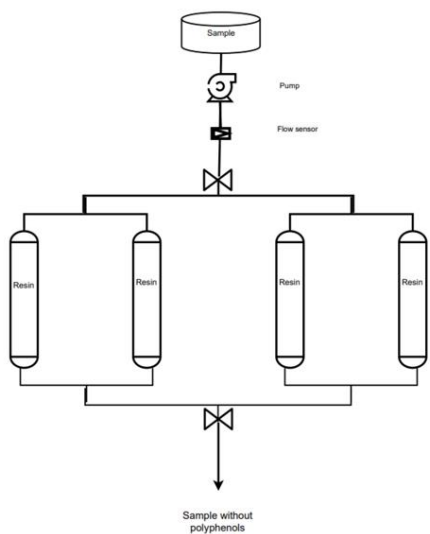
- Static extraction experiments were performed employing hot water and organic solvents
- Water-methanol mixture (50:50 b.v.) yielded **69% polyphenols recovery**
- Currently working on dynamic extraction experiments,
- Aiming to optimise:
 - experimental conditions and
 - solvent recovery and reuse strategy



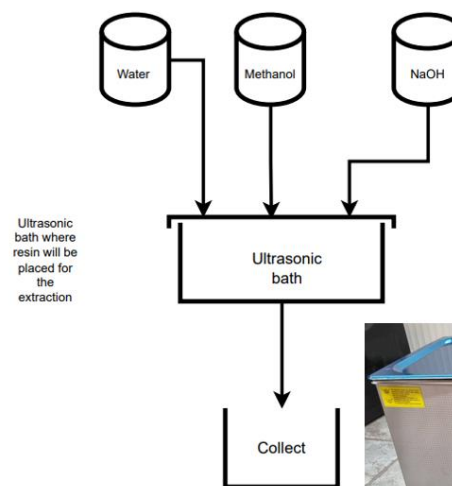


Design and construct the pilot system

Adsorption Unit



Extraction unit





Installation of GtG Polyphenol Extraction Unit

- Installation of the polyphenol adsorption unit at Karmiel wastewater treatment plant
- Integration with the polyphenol adsorption unit with the high-rate anaerobic reactor
- The unit has been operating since December 2022



27



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Polyphenol Extraction Testing



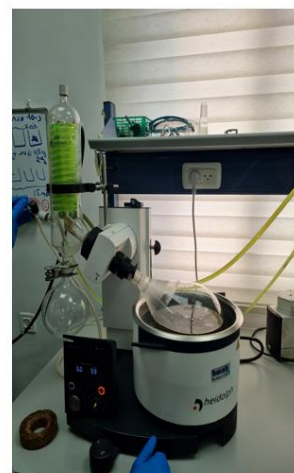
Adsorption steps:

- Wastewater from olive oil mills is passed through the resin- packed bed
- Polyphenols are selectively adsorbed on the resin
- The resins are removed and transferred to the lab for testing





Polyphenol Extraction Testing



Liquid-solid extraction of polyphenols in the lab at the Galilee Society with methanol/water as the solvent





CS6: Task 1.4.5 – Timeline- Karmiel

Subtask: 1.4.5 Recovery of high-value products from olive mill wastewater in Karmiel



T1.4.5 - Recovery of high-value products from olive mill wastewater in Karmiel

Baseline conditions assessed

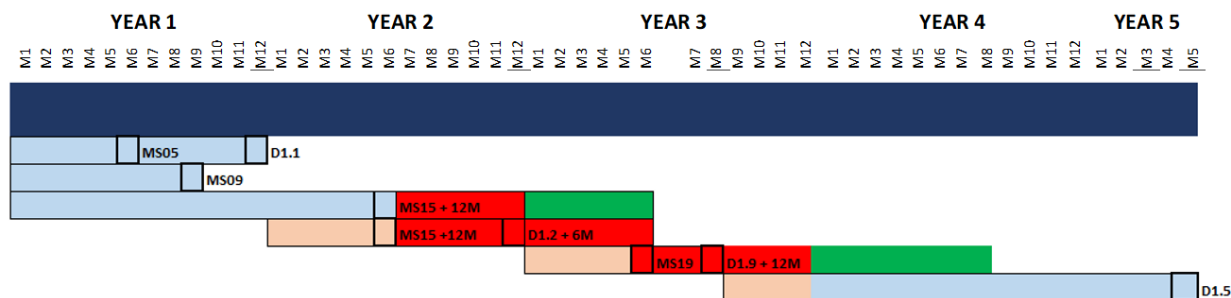
Design of pilot system

Bench scale experiments

Pilot system operational

Start-up & results

Best practices for material recovery



→ Pilot system start-up took place in December 2022

30



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Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity
- Extension of activity



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CS6 Contacts

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khalid@gal-soc.org



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2.7. CS7: Tain

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
7	1.2.6	AnMBR + RO	50%	100%	100%	Aug 22
	1.3.5	AnMBR + heat utilisation from its effluent		100%	100%	Aug 22
	1.4.6	Recovery of ammonia via stripping	>100%	100%	100%	Sep 22

ULTIMATE



WATER SMART INDUSTRIAL SYMBIOSIS

D1.2 Operational demo cases

CS7 Tain

UCRAN, Aquabio





CS7: Tain

Lead partner:



Other partner:



With support of:



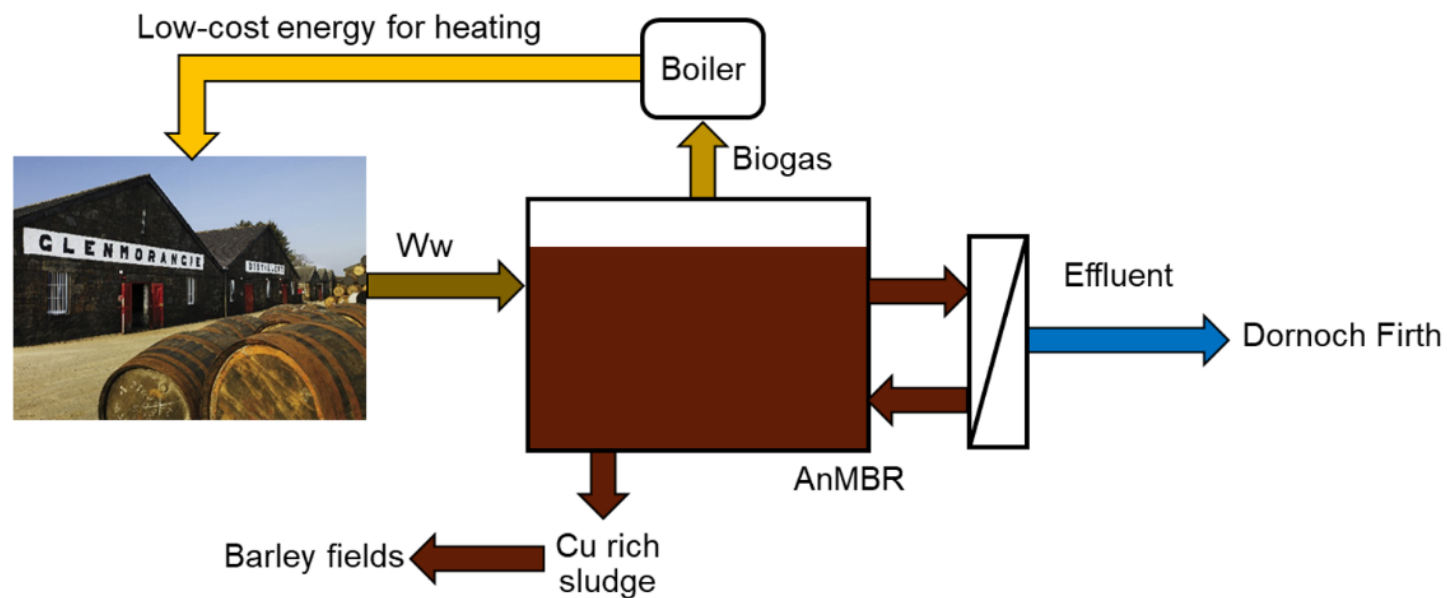
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2





CS7: Situation before Ultimate



3



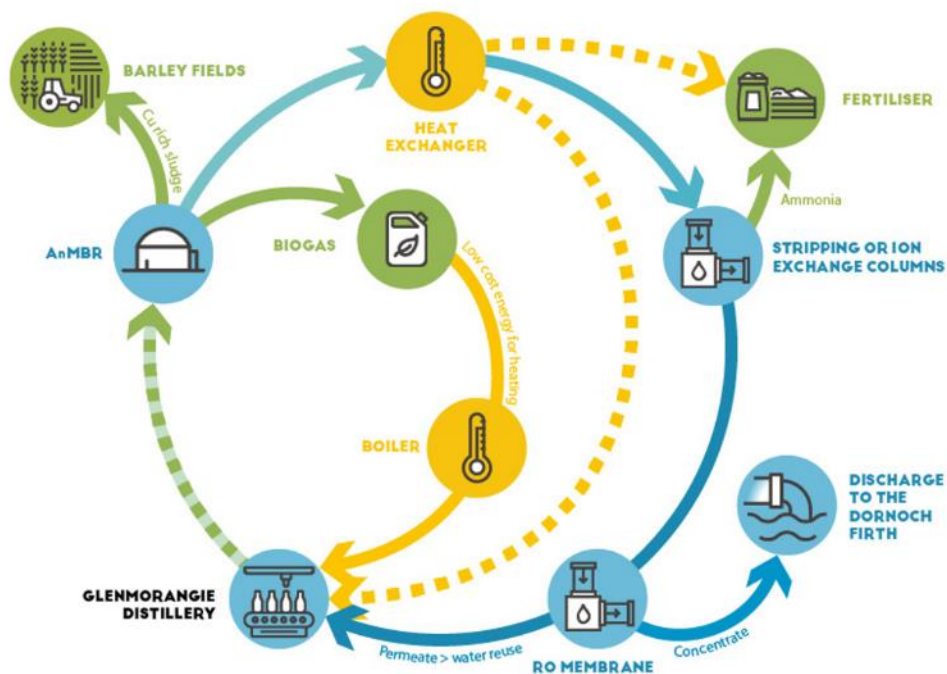
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CS7: Objectives of the Ultimate solutions



4



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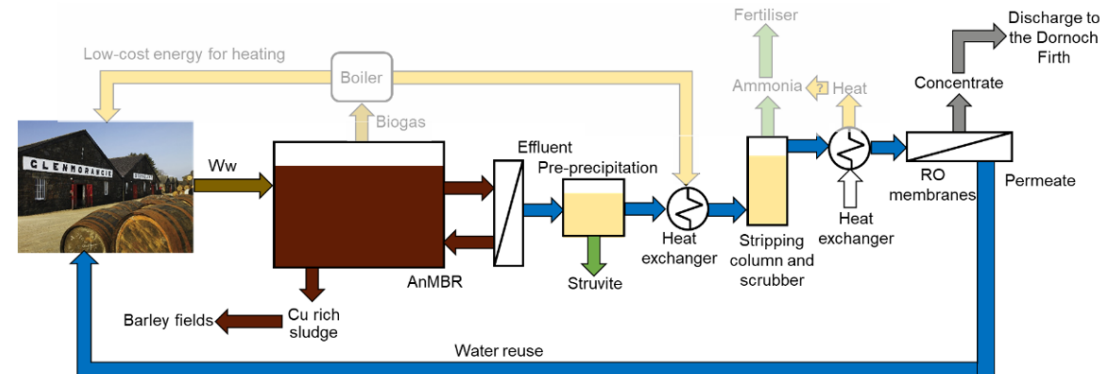


CS7: Subtask 1.2.6 status/progress

Subtask: 1.2.6 RO treatment of distillery wastewater after AnMBR for internal water reuse

Baseline technology: no water reuse so far (discharge of AnMBR effluent to Dornoch Firth)

Ultimate solution to foster circular economy: RO system for distillery wastewater (AnMBR effluent)



TRL: 5 → 7

Capacity of demo plant: 1 m³/d

Quantifiable target: At full scale, potential for the production of 58,000 m³/a for internal water reuse; >40 % reduction of freshwater through reuse of treated water

Status/progress:

- System operational and initial trials conducted



Horizon 2020 research and innovation programme under grant agreement No 869318





CS7: Results of the laboratory experiments

Subtask: 1.2.6 RO treatment of distillery wastewater after AnMBR for internal water reuse

Lab-scale trials were carried out to evaluate the impact of the reverse osmosis membranes position in the treatment train. Experiments with the different waters (anaerobically treated = raw, after precipitation and after stripping) were performed in a Sterlitech HP4750 lab-scale dead-end filtration cell using Trisep X201 RO flat sheet membranes to achieve 50% permeate recovery.

Anaerobic MBR effluent

Parameter	Value
pH	7.2 ± 0.3
Electrical Conductivity	6.0 ± 0.5 mS/cm
COD (Chemical Oxygen Demand)	554 ± 195 mg/L
TN (Total nitrogen)	804.7 ± 130.0 mg/L
TAN (Total ammoniacal nitrogen)	800.8 ± 96.0 mg/L
Phosphate	209.2 ± 24.1 mg/L



Reverse osmosis permeate

	Raw AnMBR Effluent	Raw AnMBR Effluent at pH 6.5	Precipitated AnMBR Effluent at pH 6.5	Stripped AnMBR Effluent at pH 6.5	Precipitated & Stripped AnMBR Effluent at pH 6.5
pH	8.6 ± 0.1	8.5 ± 0.3	7.6 ± 0.1	8.1 ± 0.1	8.1 ± 0.1
EC (µS/cm)	654 ± 7.8	524 ± 68.6	535 ± 16.3	1001 ± 200.1	926 ± 133.2
COD (mg/L)	<25	<25	<25	<25	<25
TN (mg/L)	111.0 ± 13.4	65.0 ± 8.7	70.0 ± 60.8	7.7 ± 0.0	6.9 ± 0.6
TAN (mg/L)	108.7 ± 15.4	53.6 ± 7.8	65.0 ± 1.2	2.5 ± 0.4	4.8 ± 0.4
Phosphate (mg/L)	4.8 ± 1.6	2.9 ± 1.0	0.7 ± 0.4	7.8 ± 2.7	0.4 ± 0.1

Reverse osmosis concentrate

	Raw AnMBR Effluent	Raw AnMBR Effluent at pH 6.5	Precipitated AnMBR Effluent at pH 6.5	Stripped AnMBR Effluent at pH 6.5	Precipitated & Stripped AnMBR Effluent at pH 6.5
COD (mg/L)	984 ± 36	1065 ± 178	1174 ± 9	1389 ± 127	1178 ± 59
TAN (mg/L)	1379.1 ± 30.7	1468.0 ± 80.6	1273.3 ± 64.0	12.8 ± 1.1	46.0 ± 0.7
Phosphate (mg/L)	326 ± 0.0	346.7 ± 8.1	25.3 ± 2.3	414.0 ± 11.0	23.6 ± 1.4

The RO permeate obtained from the water pre-treated through precipitation and stripping delivers the best quality for reuse. However, the RO concentrate obtained directly from the treatment of the anaerobic MBR effluent provides a more concentrated source of nutrients in a smaller volume which would make the nutrients recovery step more sustainable.

6



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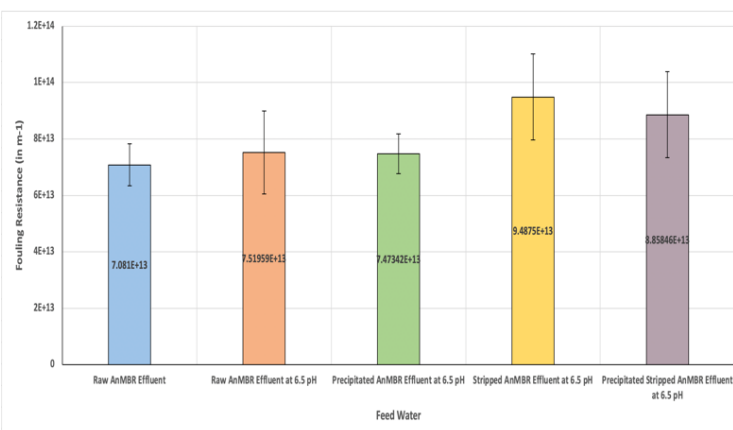
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CS7: Results of the laboratory experiments

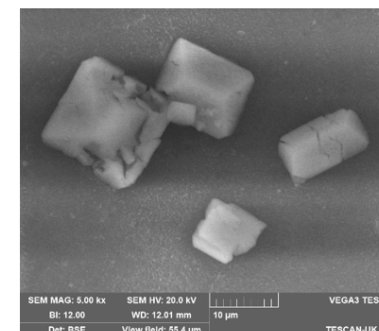
Subtask: 1.2.6 RO treatment of distillery wastewater after AnMBR for internal water reuse

Experimental trials on different feed waters in a Sterlitech HP4750 lab-scale dead-end filtration cell using Trisep X201 RO flat sheet membranes to achieve 50% permeate recovery.



Interestingly, the fouling resistance in the RO membranes was found to be slightly higher with the water which went through stripping first due to the increase in salts after pH adjustment.

However, due to the nature of the water, the effluent from the anaerobic MBR (labelled raw here) produced a more complex fouling with struvite crystals identified on the surface of the membrane.



Overall, pH adjustment (increase for the precipitation and stripping steps and decrease for the RO filtration) significantly increases the salt concentration in the water.

The sequence of the technologies in the treatment train can be adapted but it will lead to trade-offs between membrane fouling, resource recovery potential and quality of water for reuse.





CS7: Pictures and initial results of the technology

Subtask: 1.2.6 RO treatment of distillery wastewater after AnMBR for internal water reuse



The RO unit is designed to achieve high quality water for reuse from the distillery wastewater after treatment through a pre-precipitation stage and ammonia stripping. The system is fitted with TriSep 1812 X20 membrane elements. Trials were so far conducted in batch to evaluate the operability of the unit to meet a water recovery of 50%.





CS7: Task 1.2.6 - Timeline

Subtask: 1.2.6 RO treatment of distillery wastewater after AnMBR for internal water reuse

T1.2.6 - RO treatment of distillery wastewater after AnMBR for internal water reuse in Tain

Baseline conditions assessed

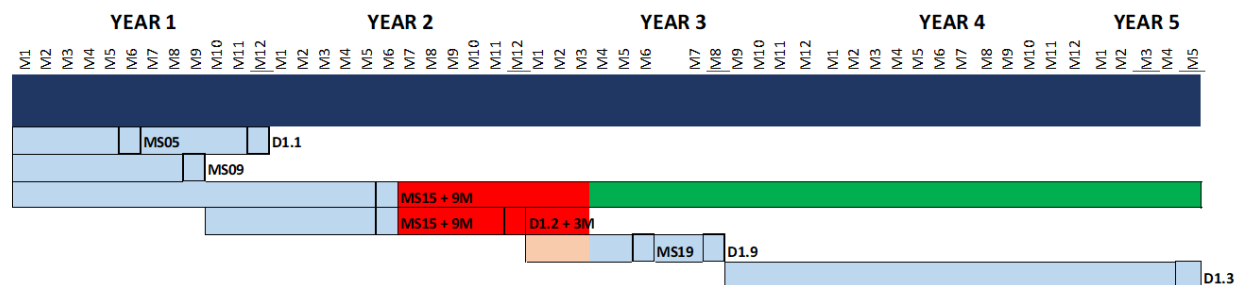
Design of pilot system

Laboratory scale experiments

Pilot system operational

Start-up & results

Best practices for water recycling



→ Pilot scale experiments started in August 2022 (M27)

→ Additional lab scale experiments will continue to be carried out in parallel to the operation of the pilot unit to help further support the evaluation

9



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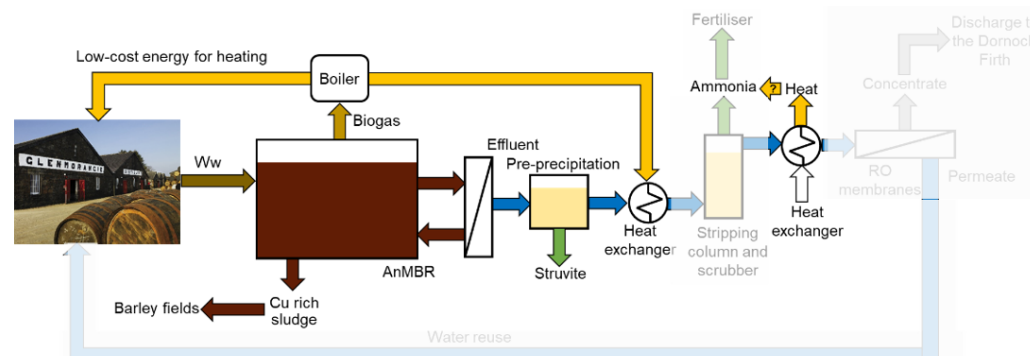


CS7: Subtask 1.3.5 status/progress

Subtask: 1.3.5 Heat recovery from treated (AnMBR) distillery wastewater

Baseline technology: Biogas production via existing AnMBR; no heat recovery before Ultimate

Ultimate solutions to foster circular economy: heat from the AnMBR effluent utilized in subsequent treatment steps



TRL: 5 → 7

Capacity of demo plant: heat utilization will be tested in all systems at 1 m³/d for the RO and 12 m³/d for the nutrients recovery system and 14 kW of heat recovery can be expected

Quantifiable targets: At full scale, >15 % reduction of energy demand from biogas and 60 % heat recovery within stripping column unit

Status/progress:

- System operational and initial trials conducted



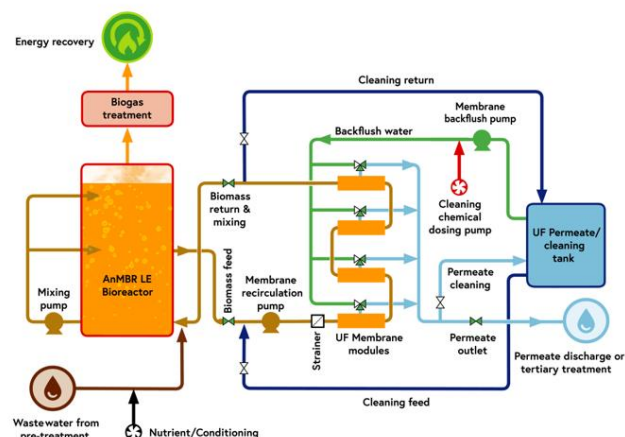
CS7: First results of the new technologies

Subtask: 1.3.5 Heat recovery from treated (AnMBR) distillery wastewater

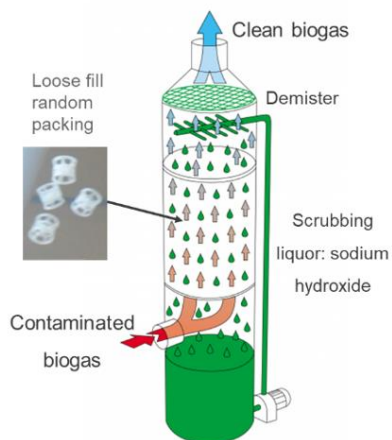
The biogas produced in the AnMBR first goes through a scrubber for H₂S removal and is then converted to steam in a boiler.

The steam produced is reused to heat the stills in the distillery and contribute to reduce its dependence on fossil fuel by 15%.

Biogas Generation (Nm ³ /d)	8,000
CH ₄ content (%)	55-70



Packed tower scrubber for H₂S removal



Biogas fired steam boiler



Maximum continuous rating (kg/hr)	2067
Design temperature (°C)	188
Working pressure	8 barg

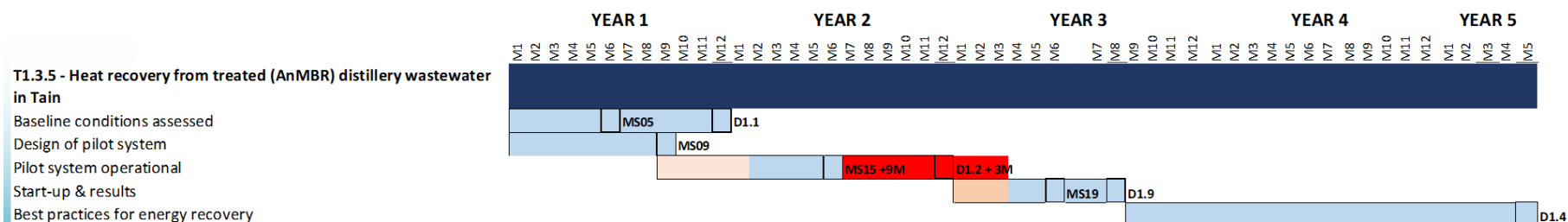
<https://www.forbesgroup.co.uk/environmental-technologies/packed-tower/>





CS7: Task 1.3.5 - Timeline

Subtask: 1.3.5 Heat recovery from treated (AnMBR) distillery wastewater



- Residual heat utilisation trials were started in August 2022 (M27) to evaluate the impact of temperature in the reverse osmosis membrane and further trials will be carried out in the nutrients recovery steps.
- Monitoring of the biogas and steam productions from the full scale AnMBR continues

Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity



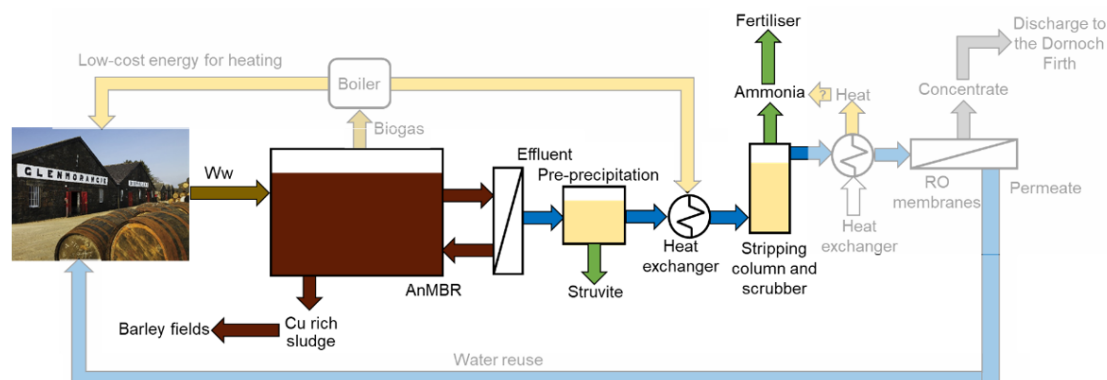


CS7: Subtask 1.4.6 status/progress

Subtask: 1.4.6 Recovery of ammonia from distillery wastewater via IEX/packed columns after AnMBR

Baseline technology: reuse of digestate on the barley fields

Ultimate solution to foster circular economy: air stripping column & scrubber; struvite precipitation



TRL: 5 → 7 (air stripping column & scrubber); 5 → 7 (struvite precipitation)

Capacity of demo plants: 12-24 m³/d

Quantifiable target: At full scale, potential for the production of 122 t struvite/a from the pre-precipitation stage and 47 t nitrogen/a from ammonia stripping, corresponding to about 80% P recovery and 80% N recovery in total

Status/progress:

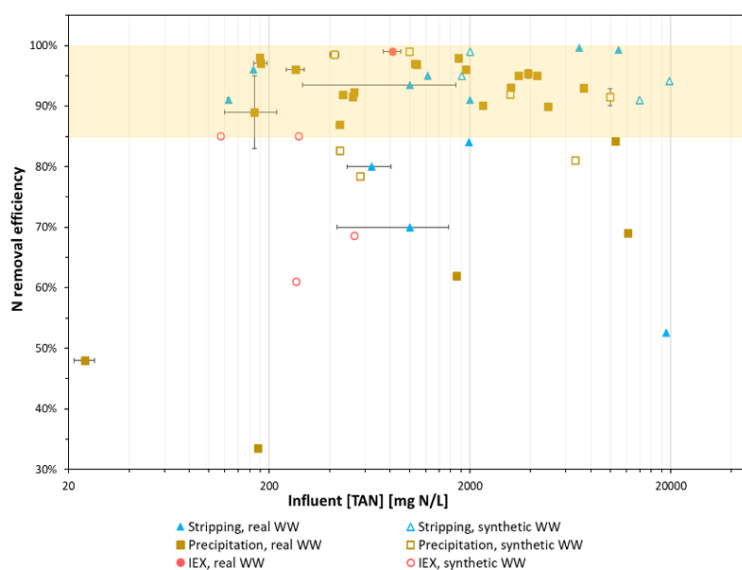
- System operational and initial trials conducted





CS7: Results of the preliminary evaluation

Subtask: 1.4.6 Recovery of ammonia from distillery wastewater via IEX/packed columns after AnMBR



The evaluation of current knowledge and performance of ion exchange, stripping and precipitation based systems for ammonia recovery from industrial wastewaters and the measured characteristics of the anaerobically treated distillery wastewater led to the selection of a two-stage system comprising pre-precipitation (struvite) followed ammonia stripping to maximize the recovery of nutrients.





CS7: Pictures of the struvite precipitator and ammonia stripping unit

Subtask: 1.4.6 Recovery of ammonia from distillery wastewater after AnMBR



15



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CS7: Task 1.4.6 - Timeline

Subtask: **1.4.6 Recovery of ammonia from distillery wastewater after AnMBR**

T1.4.6 - Recovery of ammonia from distillery wastewater by IEX/packed columns after AnMBR in Tain

Baseline conditions assessed

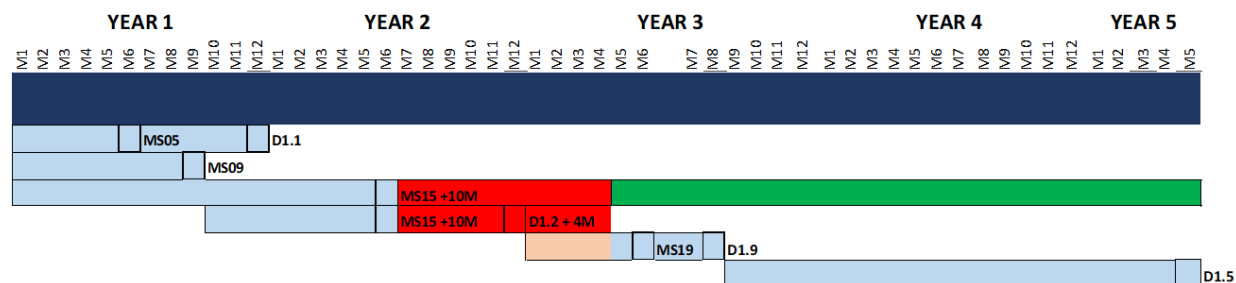
Design of pilot system

Laboratory scale experiments

Pilot system operational

Start-up & results

Best practices for material recovery



→ Nutrients recovery systems were commissioned in September 2022 (M28).

→ The pilot and lab scale experiments will continue in parallel until the end of the project

16



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Legend

Task/Subtask	
Activity as planned	
Postponed activity	
Delay of activity	
Extension of activity	



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WATER SMART INDUSTRIAL SYMBIOSIS

CS7 Contacts

m.pidou@cranfield.ac.uk

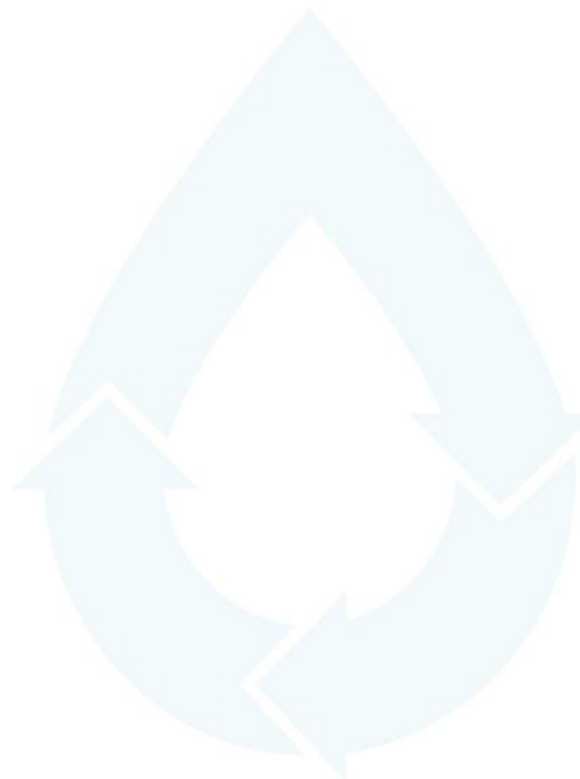
M.M.Gritti@cranfield.ac.uk



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2.8. CS8: Chemical platform of Roussillon

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
8	1.3.6	Feasibility study: heat recovery	No pilot plant --> excluded from D1.2			
	1.4.7	Laboratory pilot: sulphur recovery	90%	100%	100%	Oct. 22
		Industrial pilot: sulphur recovery		35%	0%	Aug 23
		Feasibility study: metal recovery	No pilot plant --> excluded from D1.2			



D1.2 Operational demo cases

CS8 Chemical platform Roussillon

SUEZ RR, 3S



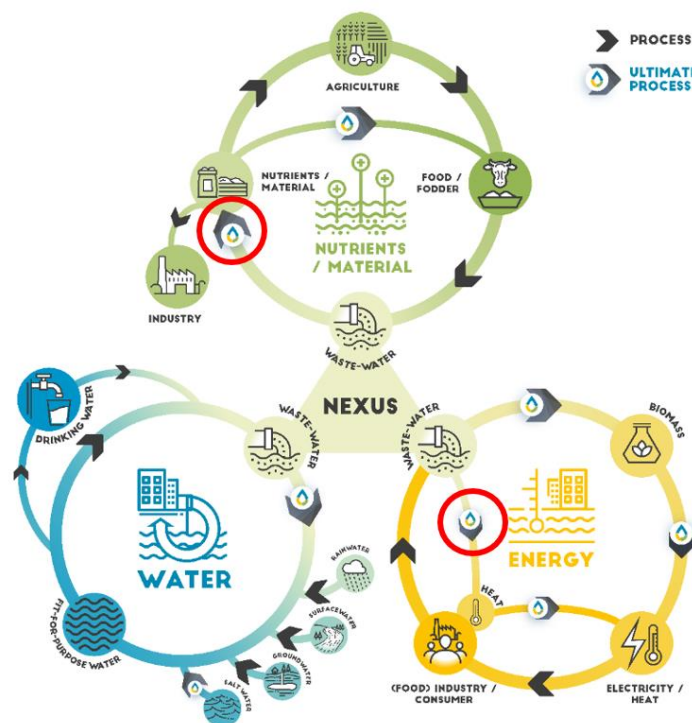


CS8: Chemical platform of Roussillon

Lead partner:

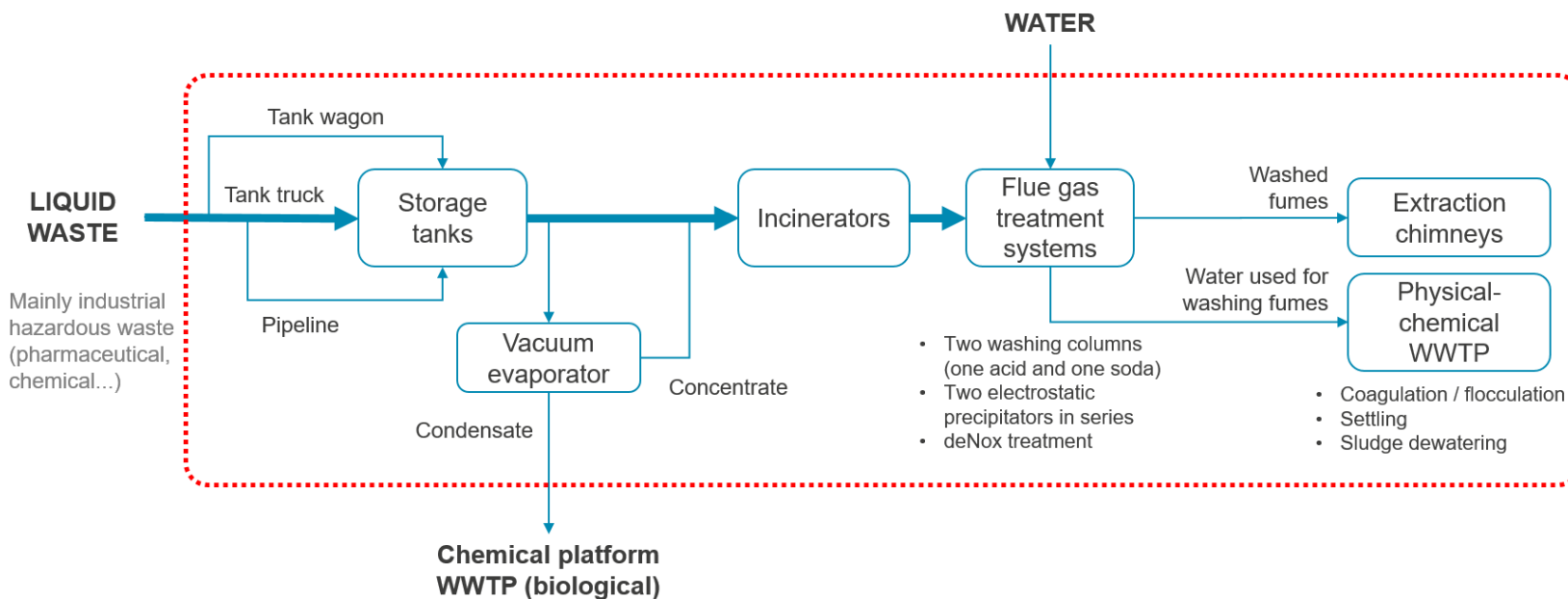


Other partner:



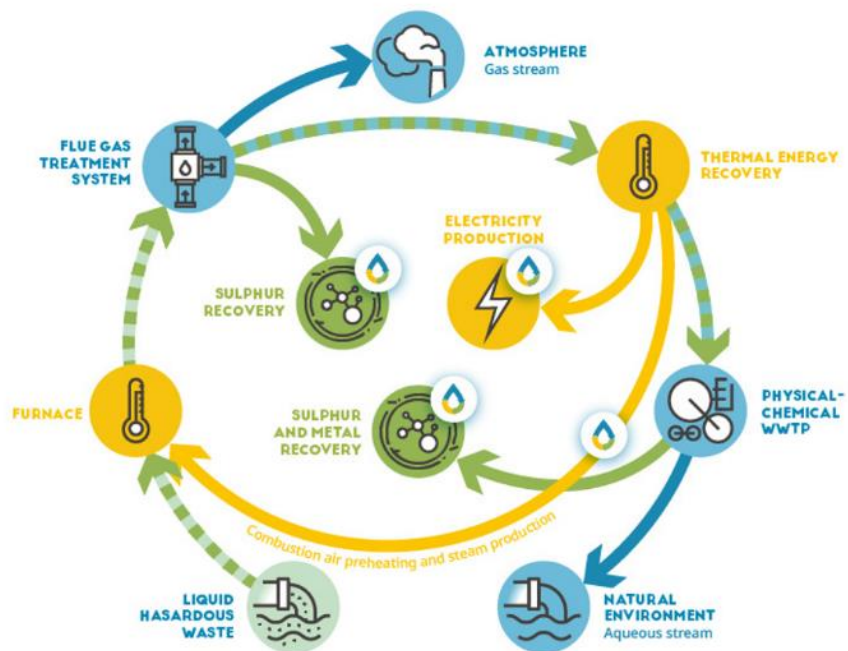


CS8: Situation before Ultimate





CS8: Objectives of the Ultimate solutions



4



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CS8: Status/progress

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon

Baseline technology: no Sulphur nor metals recovery so far

Ultimate solution to foster circular economy:

- **Sulphur recovery from flue gas:** condensation, dust cleaning and scrubbing
- **Sulphur recovery from effluent WWTP:** electrolytic oxidation or natural flocculating agents or chemical precipitation of sulphates
- **Metals recovery:** concept study and physical-chemical modelling (calculation of metal speciation, solubility equilibria, complexation reactions).

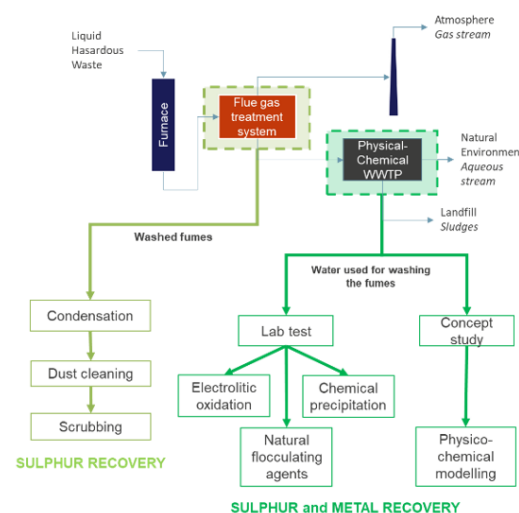
TRL: 4 → 6 (Sulphur recovery); **2 → 4** (metals recovery)

Capacity: Sulphur from flue gas: 25 000 Nm³ flue gas / h at 0 to 1% SO₂ depending on the feed waste ;
Sulphur from effluent WWTP: 1 100 m³/d corresponding to about 15 t/d of sulfates ; **Metals:** 1 100 m³/d

Quantifiable target: Sulphur from flue gas: 80% Sulphur recovery; **Sulphur from effluent WWTP:** 80% Sulphur recovery

Status/progress:

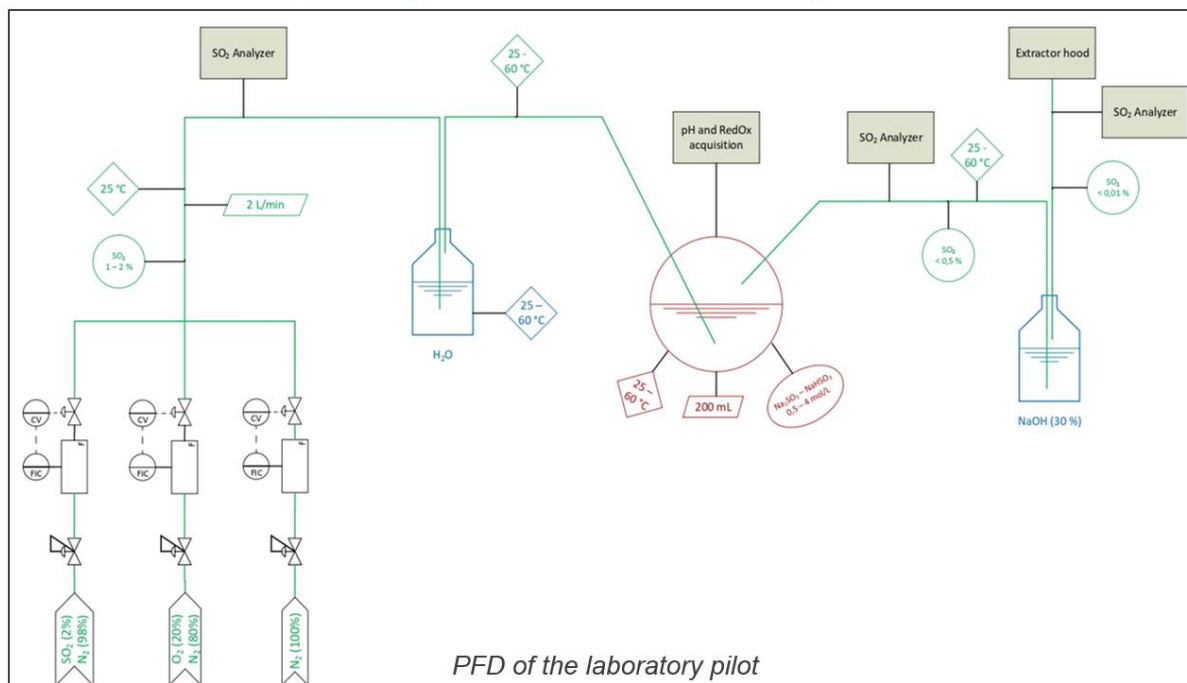
- **Sulphur from flue gas:** Laboratory pilot plant is operational & industrial pilot is under construction
- **Sulphur from effluent WWTP:** The lab tests will start in M37
- **Metals:** this task will be conducted on another plant of IWS Chemicals (Le Pont-de-Claix) and started in M34





CS8: The laboratory pilot

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon



Sizing :

Creation of a laboratory pilot able to study the impact of certain operating characteristics on the absorption of SO₂.

→ Use of an experimental design to effectively analyze these impacts.

Objective :

Determine precisely the ideal configuration to absorb SO₂ and concentrate the solution of interest.





CS8: The laboratory pilot

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon

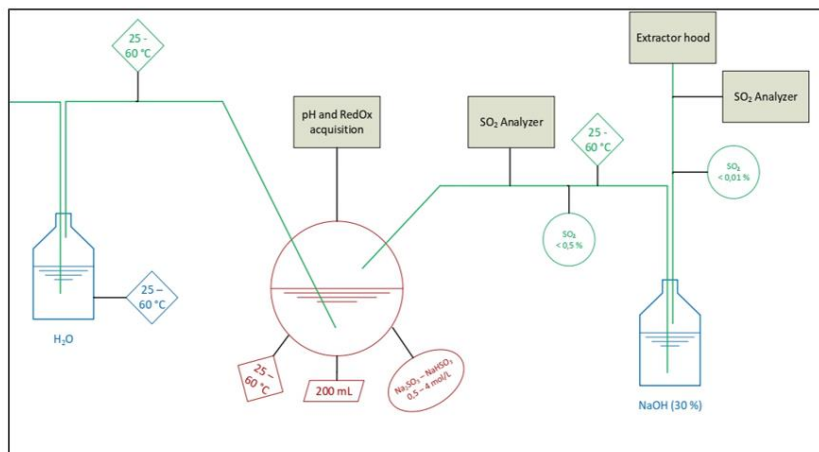
Gas mixture :

Allows to study :

- The SO_2 level (0,1 to 1 %) ;
- The O_2/SO_2 ratio (5 to 20).

Analysis :

- SO_2 analyzer ;
- Test kits (liquid phase characterization) ;
- pH sensor ;
- RedOx potential sensor.



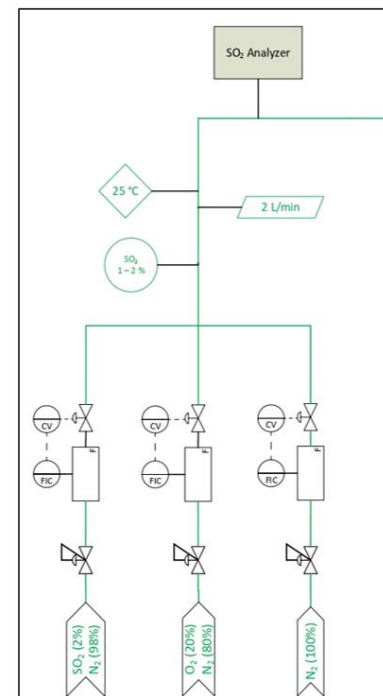
PFD of the laboratory pilot : Focus on the reactor



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Other parameters :

- Temperature (40 to 60 °C) ;
- Initial composition of the liquid phase (0,1 to 1,5 mol/L Na_2SO_3).



PFD of the laboratory pilot :
Focus on the gas mixture





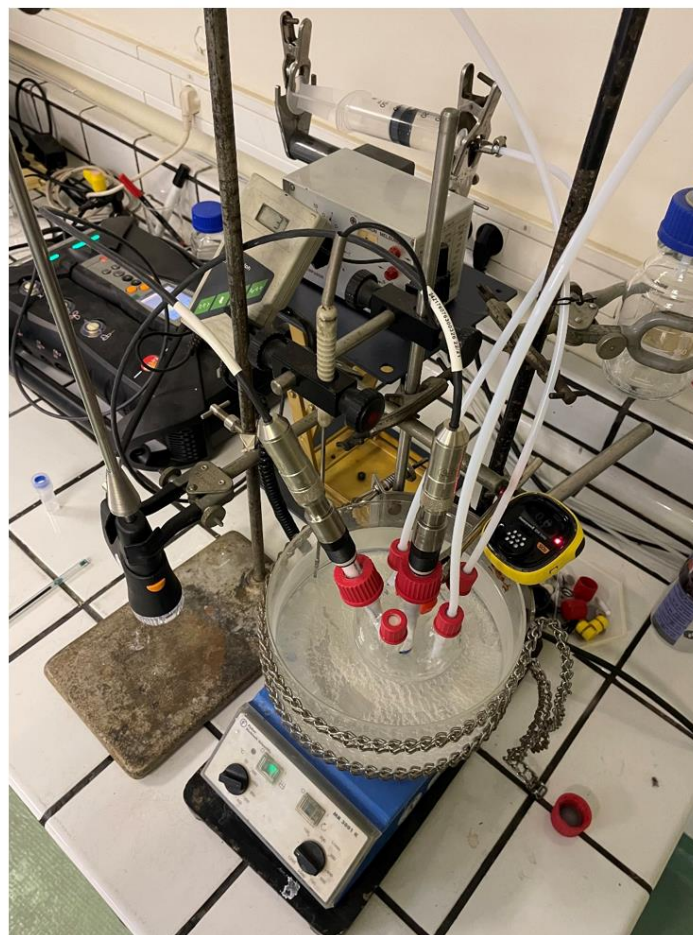
CS8:

**picture of
laboratory pilot
to recover sulphur
from flue gas**

8



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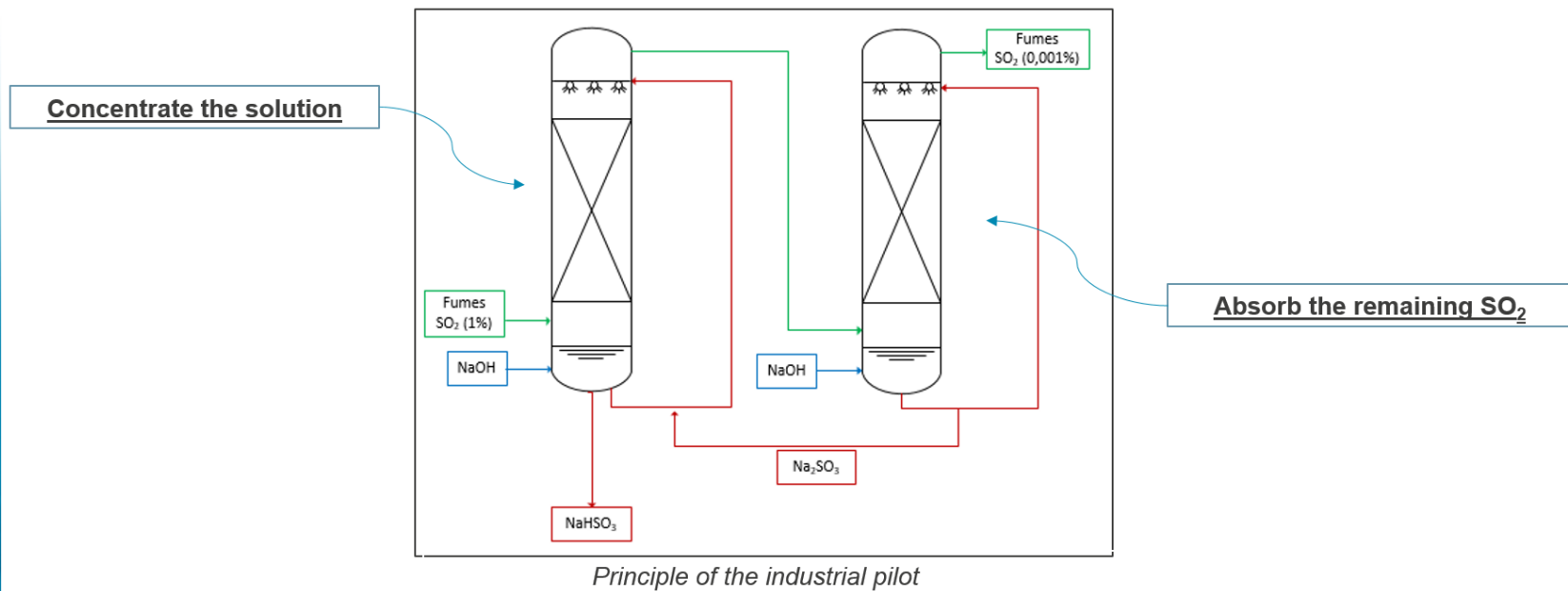


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CS8: The industrial pilot

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon





CS8: The industrial pilot

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon

Sizing :

- Column sizing ;
- Realization of the PFD and the mass balance ;
- Realization of the PID.

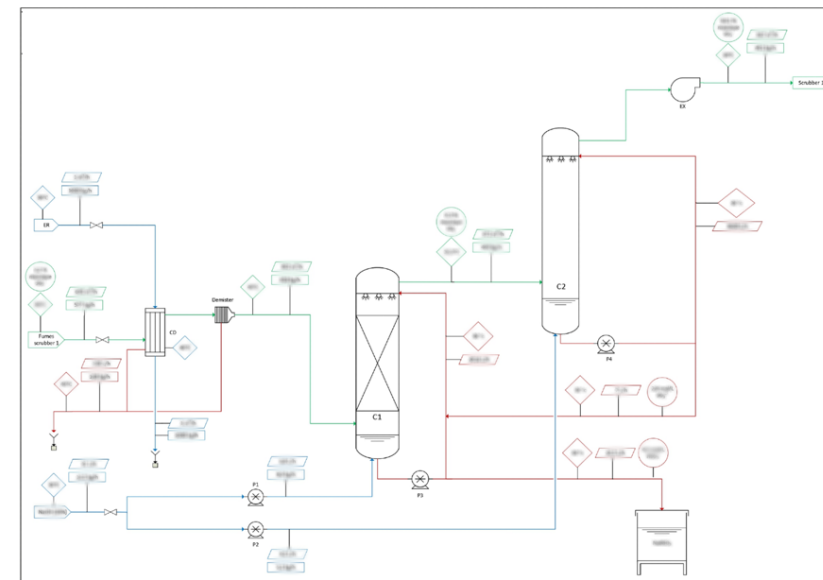
Pilot's specifications are already realized and contact with suppliers is underway.

Addition of a condenser : Required if we want to concentrate the product.

- By temperature decrease in the columns, water contained in the fumes will condense and significantly dilute the solution.

Two different columns : A packed and a spray column.

- Interesting to compare because in this case, they seem to have equivalent performances.



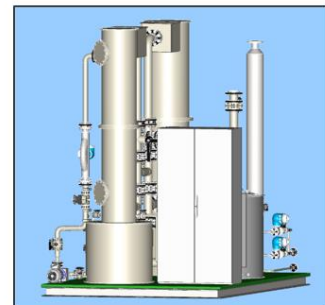
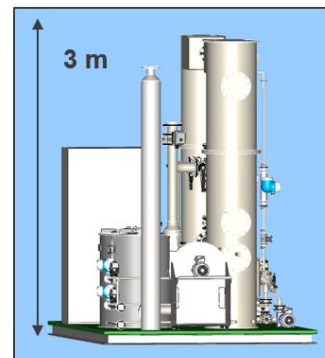
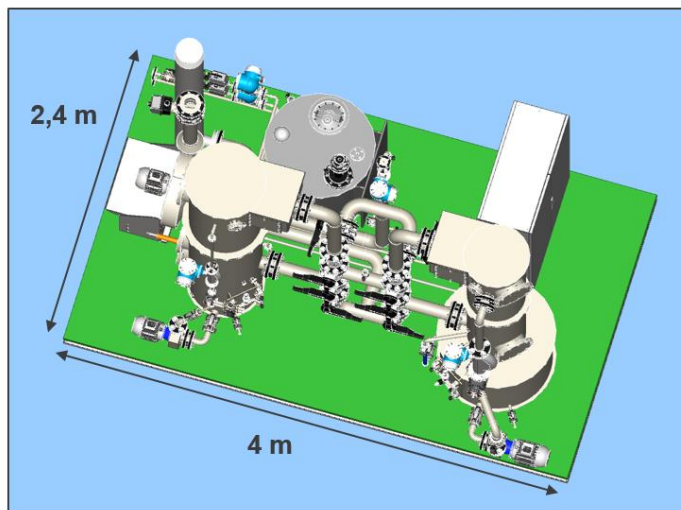
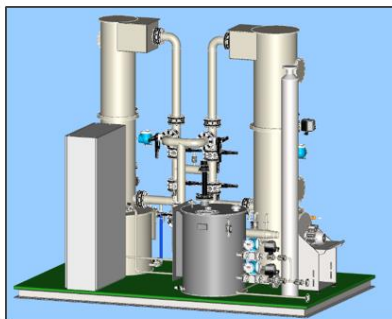
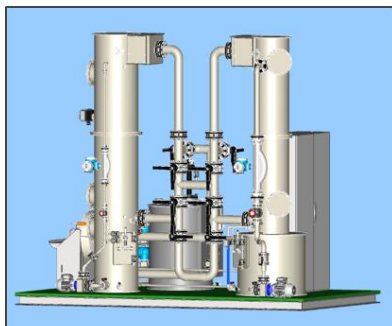
PFD of the industrial pilot





The industrial pilot – 3D model

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon



11



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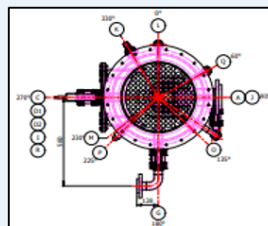
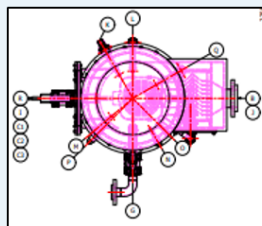
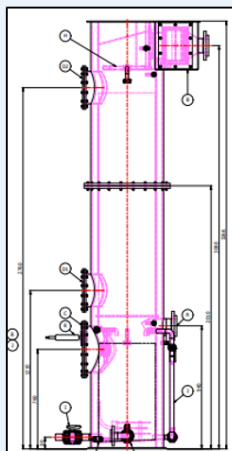
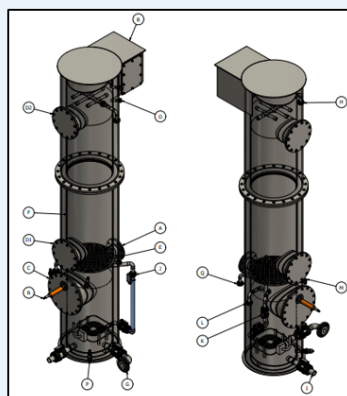
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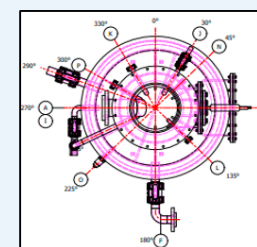
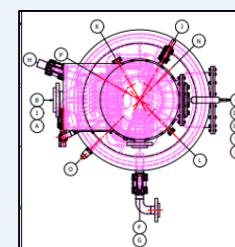
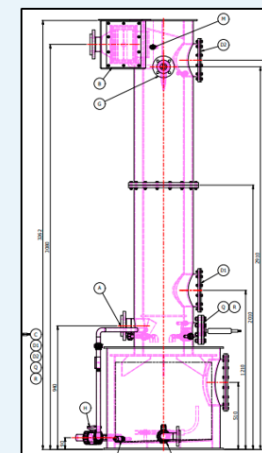
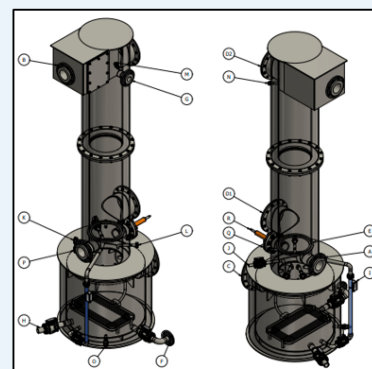
The industrial pilot – Scrubbers

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon

Scrubber D210



Scrubber D310





The industrial pilot – Manufacture

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon

Scrubber D210
(Lower part)

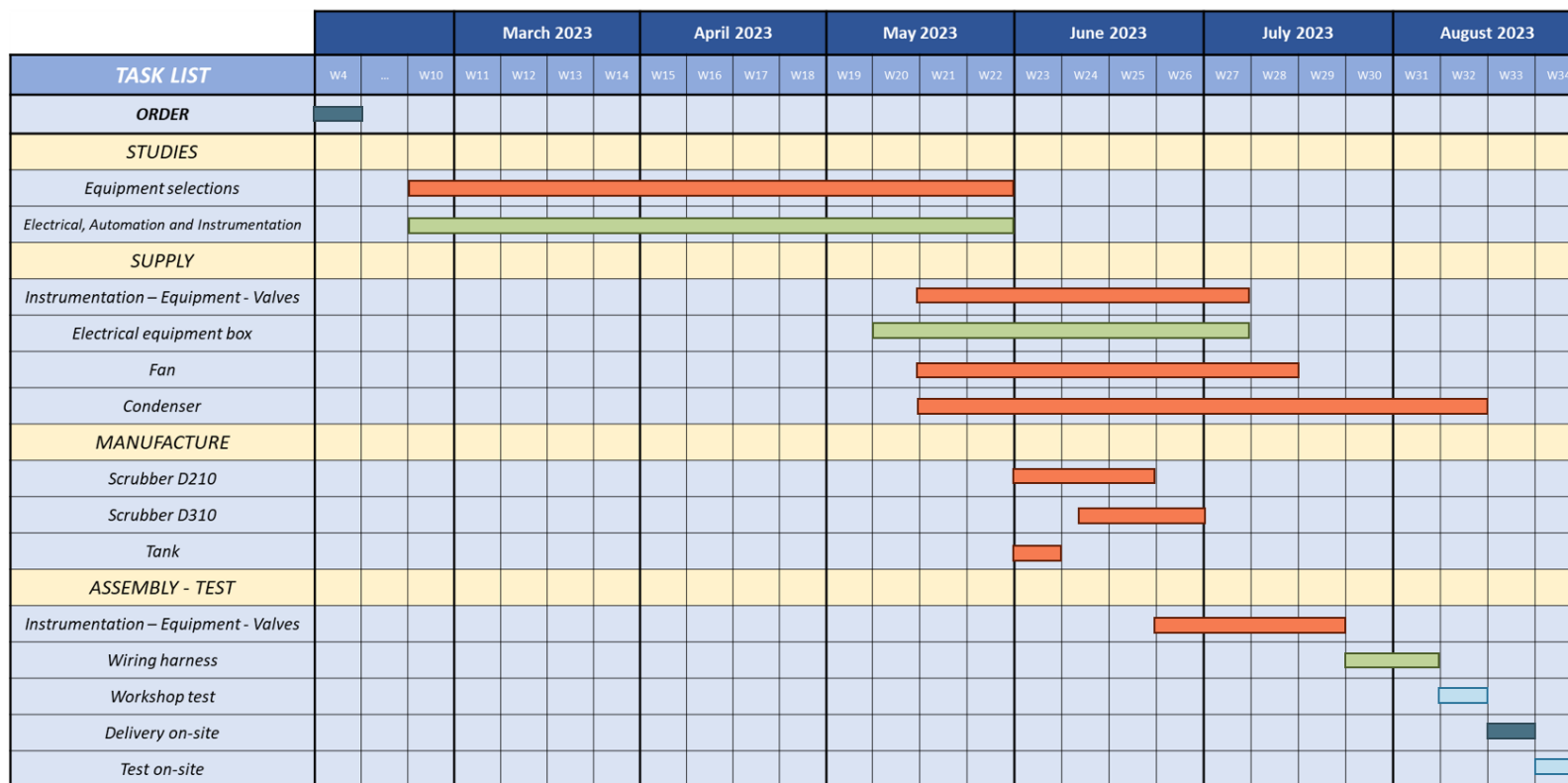


Scrubber D310
(Lower part)





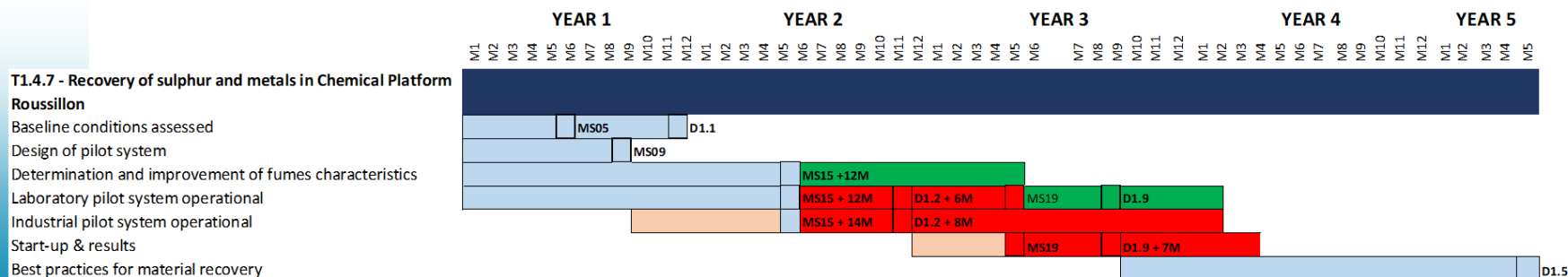
The industrial pilot – Schedule





CS8 Timeline

Subtask: 1.4.7 Recovery of Sulphur and metals at the chemical platform of Roussillon



- Laboratory pilot system is operational since Oct. 2022
- Industrial pilot plant is expected to be operational : August 2023 (M39)
- Still enough time to conduct the planned investigations in the industrial pilot



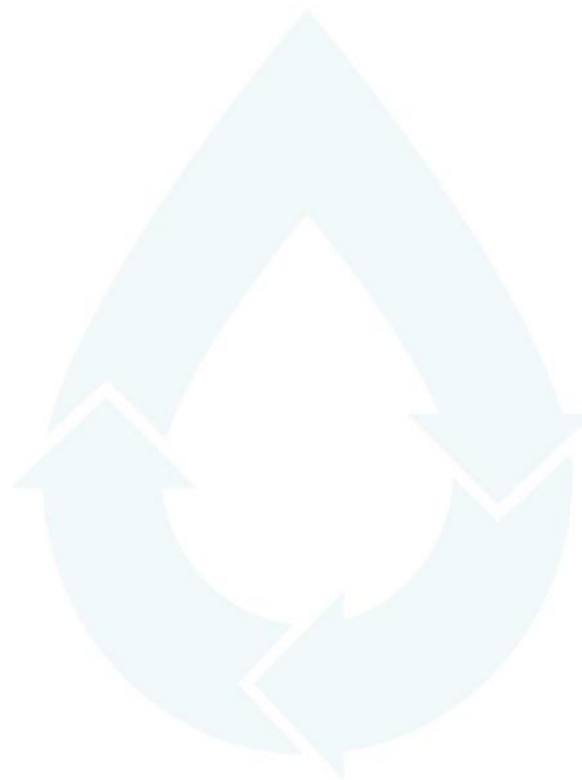


WATER SMART INDUSTRIAL SYMBIOSIS

CS8 Contacts

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2.9. CS9: Kalundborg

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
9	1.2.7	Novel UF membrane		100%	100%	Jun 21
	1.3.7	Joint control system	No pilot plant --> excluded from D1.2			
	1.4.8	Concept study: high added value product recovery	No pilot plant --> excluded from D1.2			



D1.2 Operational demo cases CS9 - Kalundborg

KWB, Kalundborg Forsyning, Novozymes, Pentair X-Flow





CS9: Kalundborg

Lead partner:  KALUNDBORG
FORSYNING

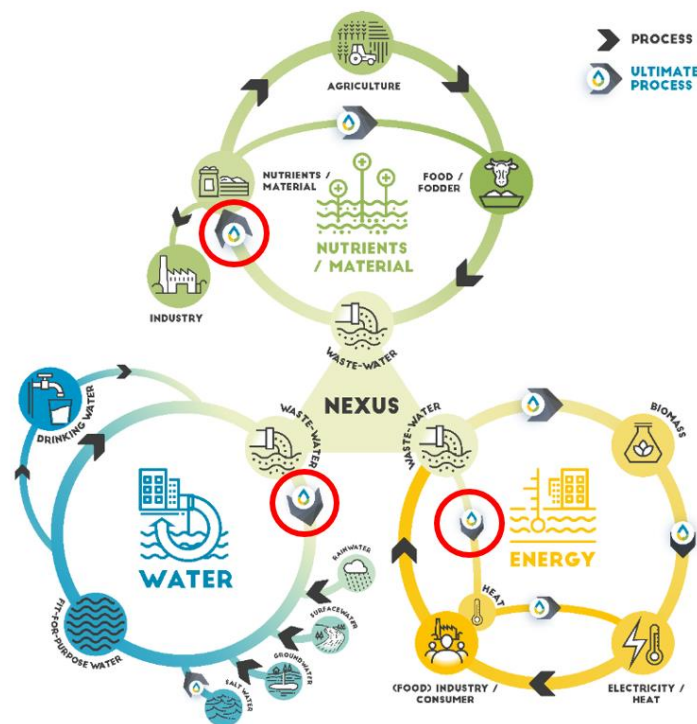
Other partners:  PENTAIR X-FLOW

 KWB
 novozymes®
Rethink Tomorrow

2



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CS9: Situation before Ultimate

- No water reclamation from WWTP effluent
- Each WWTP has its separate control system
- No high added value product recovery from wastewater so far

Kalundborg municipality



Novozymes WWTP



40%

55%

Other industries



5%



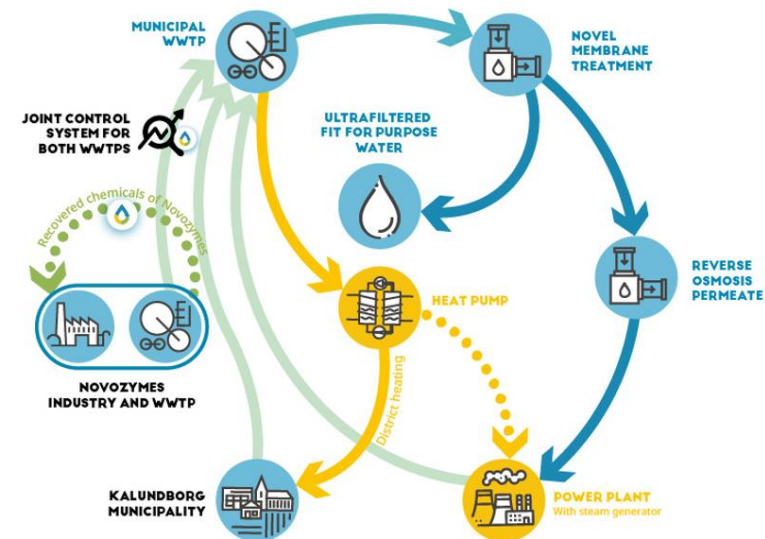
Kalundborg WWTP





CS9: Project objectives in Kalundborg:

- **Production of fit-for-purpose water** using a novel membrane pre-treatment for wastewater with high a fraction of non-degradable organic matter
- **Energy efficiency increase** through a synergetic operation of two WWTPs and concept study for heat recovery
- Concept study for **nutrient and/or high-value product recovery**



4



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CS9: Pilot plant: fit-for-purpose water production

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse

Baseline technology

- no water reuse so far (discharge to the recipient)

Ultimate solution to foster circular economy

- nanofiltration (NF) or novel ultra-tight ultrafiltration (UF) as pre-treatment for reverse osmosis (RO)

TRL 5 → 7

Capacity 2 m³/h

Ambition beyond the project

- > 70,000 m³/a fit-for-purpose water production
- > 40% reduction of surface water through reuse of treated water

Status/progress

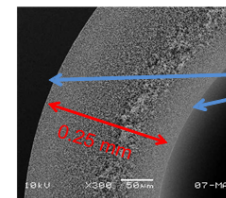
- Pilot plants in operation since June 2021



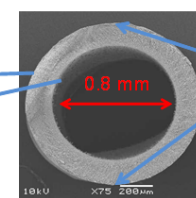
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Novel ultra-tight ultrafiltration membrane

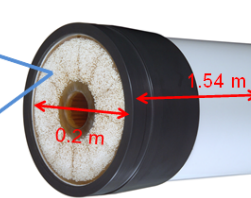
- Molecular weight cut off: 4 kDa
- Filtration: inside out



Fibre wall

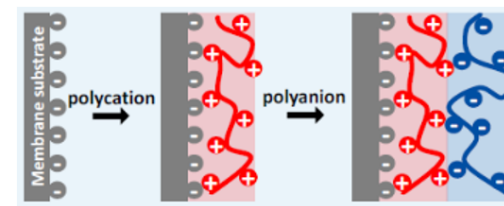


Fibre



Module head

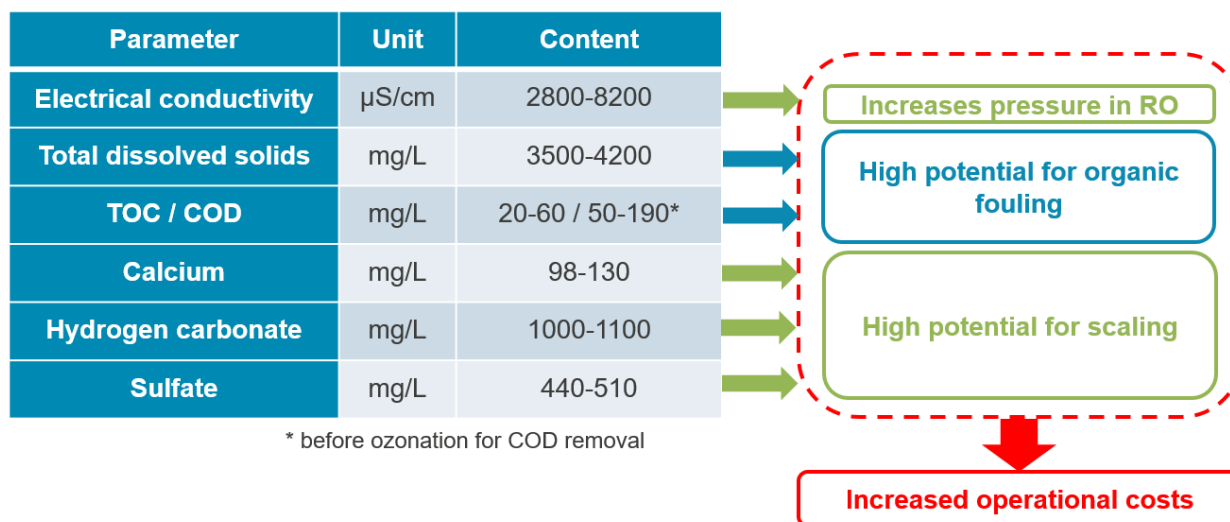
- Material: (modified) polyethersulphone with LbL





CS9: Specific challenges at the municipal WWTP

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse



6



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CS9: Operational procedures and methodologies

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse

Fouling prevention:

- Does the novel ultra tight membrane or nanofiltration membrane prevent better the RO from fouling than a conventional UF?
- Conventional UF membrane, novel UF membrane and nanofiltration membrane are operated in parallel in order to compare their performance in terms of fouling prevention

Production of fit-for-purpose water:

- Can we produce fit-for-purpose water for cooling towers and/or boilers?
- Which water quality is reached after UF and for which reuse purpose can the water be used (truck or street cleaning)?
- Investigation of water quality after each treatment step

7



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CS9: Treatment trains for water recovery

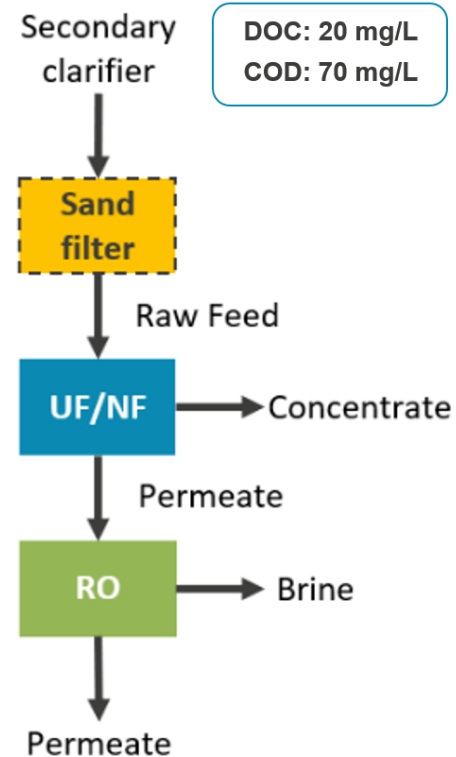
COD	Chemical oxygen demand
DOC	Dissolved organic carbon
NF	Nanofiltration
RO	Reverse Osmosis
UF	Ultrafiltration
WWTP	Wastewater treatment plant

8



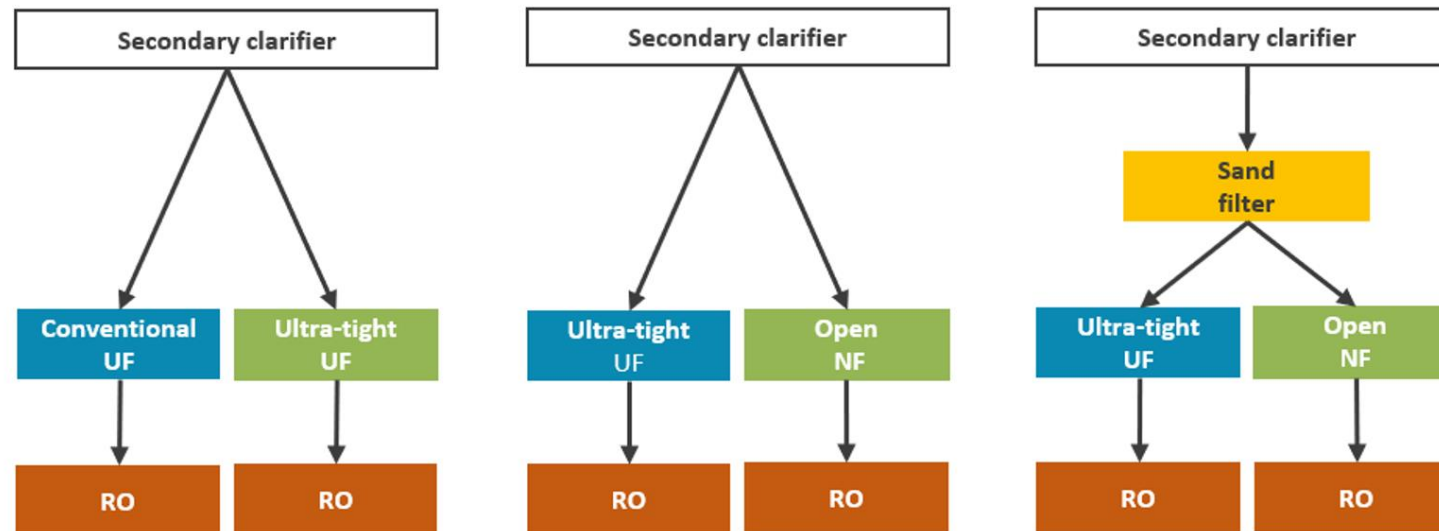
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Effluent from municipal WWTP:





CS9: Different variants of tested treatment trains





CS9: Pictures of the pilot plants

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse

Pilot A: conventional UF & RO



Pilot B: novel ultra tight UF membrane & RO



10



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CS9: Pictures of the pilot plants

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse

Pilot A (conventional UF membrane)



Reverse osmosis membranes



Pilot B (novel UF membrane)



11



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CS9: Videos of the pilot plants in operation

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse

Pilot A (conventional UF membrane)



Reverse osmosis membranes



Pilot B (novel UF membrane)



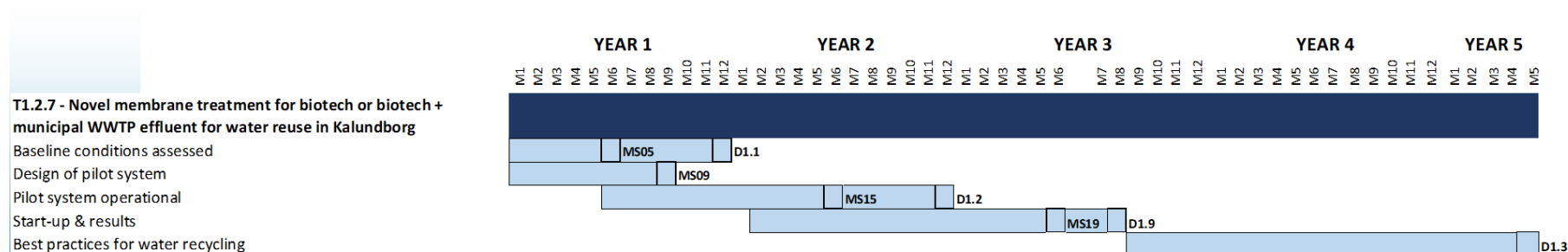
These videos are accessible via the indicated links below this presentation on the ULTIMATE webpage.





CS9: Task 1.2.7 is in time

Subtask: 1.2.7 Novel membrane treatment for biotech or biotech and municipal WWTP effluent for water reuse



- **Pilot is operational & results obtained: task is in time**
 - Due to the energy crises, the ozonation facility at the municipal WWTP was shut down. Hence, ozonation as a pre-treatment option for the water recovery treatment train cannot be investigated.
 - In addition to the foreseen Dynasand filter, a dual media filter was installed alternatively and is being investigated as pre-treatment option.

Legend

- Task/Subtask
- Activity as planned
- Postponed activity
- Delay of activity





Contacts

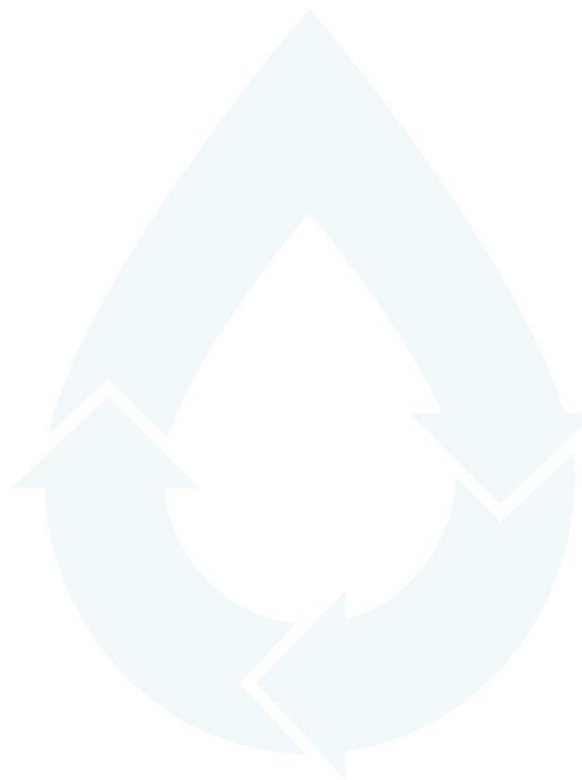
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3. Summary and conclusion

ULTIMATE aims to showcase circular economy solutions at nine case studies distributed across Europe and Israel for the treatment of industrial wastewater in order to recover water, material and energy. In this frame, 17 laboratory and preparatory experiments and investigations of existing systems are conducted to test the ULTIMATE approaches and based on them, 24 pilot plants are developed and will be demonstrated at the case studies.

Deliverable D1.2 is a demonstrator type deliverable and shows, that the ULTIMATE pilot plants are operational. To document the status for each case study, a presentation containing pictures and/or videos of the operational pilot plant is accessible on the ULTIMATE webpage (<https://ultimatewater.eu/demonstration-cases/>). This document accompanies the presentations which are meant to be the main evidence for D1.2 and shows the progress until M37.

Until M24, five pilot plants or (parts of) treatment trains were operational. Three of them are related to water recovery at the case studies in Nafplio (CS4), Lleida (CS5) and Kalundborg (CS9), one of them to material recovery in Lleida (CS5) and another one to energy recovery in Karmiel (CS6).

Until M27, five additional plants were operational. Two of them refer to water recovery in Lleida (CS5) and Tain (CS7), one to material reuse in Rosignano (CS3) and two to energy recovery and reuse in Shafdan (CS6) and Tain (CS7).

Until M30, six additional pilots were put into operation. Two relate to water recovery and are located in Tarragona (CS1). One was implemented in Nieuw Prinsenland (CS2) and focuses on both water recovery and material recovery. The other three pilots refer to material recovery and were put into operation in Nafplio (CS4), Tain (CS7) and at the Chemical Platform of Roussillon (CS8).

Since M33, four additional pilots are operational in Lleida (CS5) and in Karmiel (CS6) referring to energy recovery and material recovery, respectively.

In the course of the project, three pilots had been included in the description of work in addition. Due to their later inclusion, they have a different time planning and hence, two of them are not operational yet. They will be located in Rosignano (CS3) and are expected to be operational in September 2023 and in January 2024. Furthermore, the full-scale electrostimulated anaerobic reactor (ELSAR, CS5) has also another time planning and is expected to be operational in September 2023 as the industrial pilot plant of CS8 is at the Chemical Platform of Roussillon.

Table 3 provides an overview about the progress of the pilot systems and of the laboratory experiments. Eight WSISs conducted laboratory experiments. In total, 17 different laboratory experiments and/or investigations of already existing facilities were and are accomplished to better understand the circumstances of the real environment and to learn more about the type of technology before it is up-scaled from laboratory



to pilot scale. Eleven of the 17 investigations are already completed and four are close to be completed (75-90%).

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Table 3 Overview about the progress regarding the construction and the operation of the pilot plants: CS3 and CS5 are constructing three pilot plants that are excluded from D1.2 due to another time planning

Overview			D1.2: Operational demo cases			
CS	Subtask	Technology or treatment train	Laboratory experiments or investigations	Pilot plant constructed	Pilot plant operational	Operational since/ to be expected
1	1.2.1	RO + MD; ammonia removal via zeolites	100%	100%	100%	Nov 22
2	1.2.2	Reclamation of water using electrodialysis	75%	100%	100%	Nov 22
	1.3.1	Feasibility study: HT-ATES	No pilot plant --> excluded from D1.2			
	1.4.1	Recovery of nutrients: test beddings & demo greenhouse	75%	100%	100%	Nov 22
3	1.2.3	Control system to avoid high chlorine concentrations	No pilot plant --> excluded from D1.2			
	1.4.2	Use of byproducts: pilot scale adsorption system	100%	100%	100%	Jul 22
		Use of byproducts: clari-flocculation pilot	100%	10%	0%	Jan 24
		AOP pilot system	60%	100%	0%	Sep 23
4	1.2.4	Water recovery: filtration, AOP, SBP	100%	100%	100%	May 22
	1.4.3	Recovery of antioxidants: adsorption/extraction	100%	100%	100%	Nov 22
5	1.2.5	Monitoring system for fouling in AnMBR	No pilot plant --> excluded from D1.2			
		(NF + RO) + (AOP + UV)	100%	100%	100%	Jan./Jun. 22
	1.3.2	AnMBR	100%	100%	100%	Dec. 22
		Pilot ELSAR	100%	100%	100%	Dec. 22
		Full-scale ELSAR		50%	0%	Sep 23
		SOFC		100%	100%	Dec. 22
	1.4.4	Concept study: recovery of nutrients	No pilot plant --> excluded from D1.2			
		Solar-driven hydrothermal carbonisation demo plant	100%	100%	100%	May 22
6	1.3.3	AAT Karmiel		100%	100%	May 22
	1.3.4	AAT + membrane filtration incl. AC Shafdan	90%	100%	100%	Aug 22
	1.4.5	Recovery polyphenols (pilot system: adsorption column)	100%	100%	100%	Dec. 22
7	1.2.6	AnMBR + RO	50%	100%	100%	Aug 22
	1.3.5	AnMBR + heat utilisation from its effluent		100%	100%	Aug 22
	1.4.6	Recovery of struvite & ammonia via stripping	>100%	100%	100%	Sep 22
8	1.3.6	Feasibility study: heat recovery	No pilot plant --> excluded from D1.2			
	1.4.7	Laboratory pilot: sulphur recovery	90%	100%	100%	Oct. 22
		Industrial pilot: sulphur recovery		35%	0%	Aug 23
		Feasibility study: metal recovery	No pilot plant --> excluded from D1.2			
9	1.2.7	Novel UF membrane		100%	100%	Jun 21
	1.3.7	Joint control system	No pilot plant --> excluded from D1.2			
	1.4.8	Concept study: high added value product recovery	No pilot plant --> excluded from D1.2			

Until all pilot plants will be operational, a very close monitoring of the case studies will be done by the WP1 management team with the case study leaders and the risk officer via regularly meetings. In addition, the presentations referring to D1.2 will be further updated every three months until every pilot plant will be operational. Those results will then be reported in the best practice guidelines (D1.3, D1.4 and D1.5).



4. Literature references

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