Deliverable 5.2

Design parameters for CE processes (focus on phosphorus, water and energy recovery)

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[1] **R**=Document, report; **DEM**=Demonstrator, pilot, prototype; **DEC**=website, patent fillings, videos, etc.; **OTHER**=other

[2] **PU**=Public, **CO**=Confidential, only for members of the consortium (including the Commission Services), **CI**=Classified

Executive Summary

This deliverable comprises the description of the design of the pilot units of CS4 and CS5. The first part is focused on the pilot units of CS4. Based on the nanofiltration (NF) and reverse osmosis (RO) experiments performed at bench-scale, NF90-4040 and the LC HR 4040 membranes were selected for the pilot unit. Regarding BioPhree, this technology was already tested at pilot scale, so no bench-scale tests were needed for the design. The capacity of this unit constructed by Wetsus can be adapted for each case study (1.0 m^3 h^{-1} for CS4 and 0.4 L h^{-1} in CS5). The MED and LTE evaporator as well as the crystallizer were already constructed and tested in previous projects. NF and RO units were assembled in NTUA. All the units were transported to Larnaca (Cyprus) inside containers, in December 2021.

The second part of the deliverable is focused on the design of the units of CS5. Higher organic loading rate (OLR) was achieved with the EGSB reactor in comparison to the UASB reactor (3.9±1.3 kg COD m^{-3} d^{-1}) at bench-scale, while permeate quality and removal efficiencies were similar. Therefore, EGSB configuration was selected for the pilot unit. Considering the performance of the bench-scale reactor, a conservative value of the nitrogen loading rate (0.5 kg N m^{-3} d^{-1}) was used for the design of the PN reactor. In case of the Anammox reactor, the pilot plant was designed to apply an NLR of 0.5 kg N m^{-3} d^{-1} expecting a higher performance than the one achieved at bench-scale. Preliminary design parameters for the ViviCryst unit are also presented. The latest more important modification made by Wetsus was to remove the recirculation from the process.

Finally, some concluding remarks about the design process of the pilot units are given in the last section.

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Glossary

AnMBR	Anaerobic membrane bioreactor
AOB	Ammonia-oxidizing bacteria
AOR	Ammonium oxidation rate
BNR	Biological nitrogen removal
BOD_5	Biological oxygen demand after 5 days
CE	Circular economy
COD	Chemical oxygen demand
CIP	Cleaning in place
DO	Dissolved oxygen
EC	Electrical conductivity
EGSB	Expanded granular sludge bed
EBCT	Empty bed contact time
FA	Free ammonia
GFRP	Glass fibre reinforced polymer
LMH	$L\ m^{-2}\ h^{-1}$
LTE	Low temperature evaporator
MBR	Membrane bioreactor
MED	Multi-effect distillation
MW	Molecular weight
NF	Nanofiltration
NOB	Nitrite-oxidizing bacteria
NRE	Nitrogen removal efficiency
NLR	Nitrogen loading rate
NRR	Nitrogen removal rate
OLR	Organic loading rate

oP	Orthophosphate
P	Phosphorus
ppb	Parts per billion
RO	Reverse osmosis
SBR	Sequential batch reactor
SVI_{30}	Sludge volume index at 30 minutes
SVI_5	Sludge volume index at 5 minutes
TMP	Transmembrane pressure
TOC	Total organic carbon
TSS	Total suspended solids
UF	Ultrafiltration
UASB	Up-flow anaerobic sludge blanket
V_s	Superficial velocity
VSS	Volatile suspended solids
WW	Wastewater
WWTP	Wastewater treatment plant

1. Introduction & Objectives

The aim of work package 5 (WP5) is to demonstrate the feasibility to recover phosphorus, water, and energy from urban wastewaters. To achieve this goal, different innovative technologies will be implemented in two demonstration sites: case study (CS) 4 and 5. CS4 will be implemented in Larnaca WWTP (located in Cyprus) and CS5 in La Lagosta WWTP (Located in Barcelona, Spain). The treatment trains to be implemented in CS4 and CS5 are presented in Figure 1 and 2.

For the implementation of the prototypes, several bench-scale tests have been performed. The work and results related to these bench-scale tests were presented in the report D5.1 “Report on bench-scale tests for CS4 &CS5”. The results of these bench-scale tests served as a basis for the design and sizing of the prototypes that will be operated in both sites to demonstrate the proposed CE treatment schemes in each case study.

The objective of this report is to provide the main information about the design and sizing of the pilot units for the circular economy (CE) processes that will be operated in CS4 and CS5. Therefore, it contains the design methodology and final design parameters used for the construction of both pilot units (activities related to Task 5.1.2 Design & construction of demo plant CS4, and Task 5.1.3 Design and construction of demonstration plant for CS5).

The first part of the report presents a summary of the relevant results obtained at bench-scale in each case study, which were used for the design and sizing of the pilot units. The second part presents the design procedure and the resulting design parameters of each process unit that was constructed in each case study.

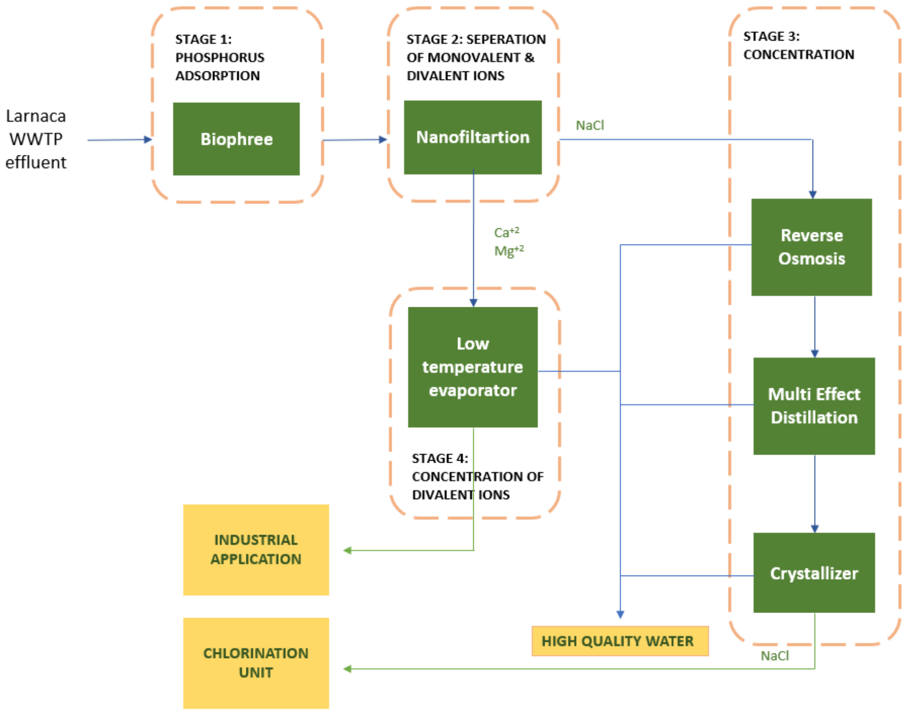

Figure 1. Process diagram for CS4

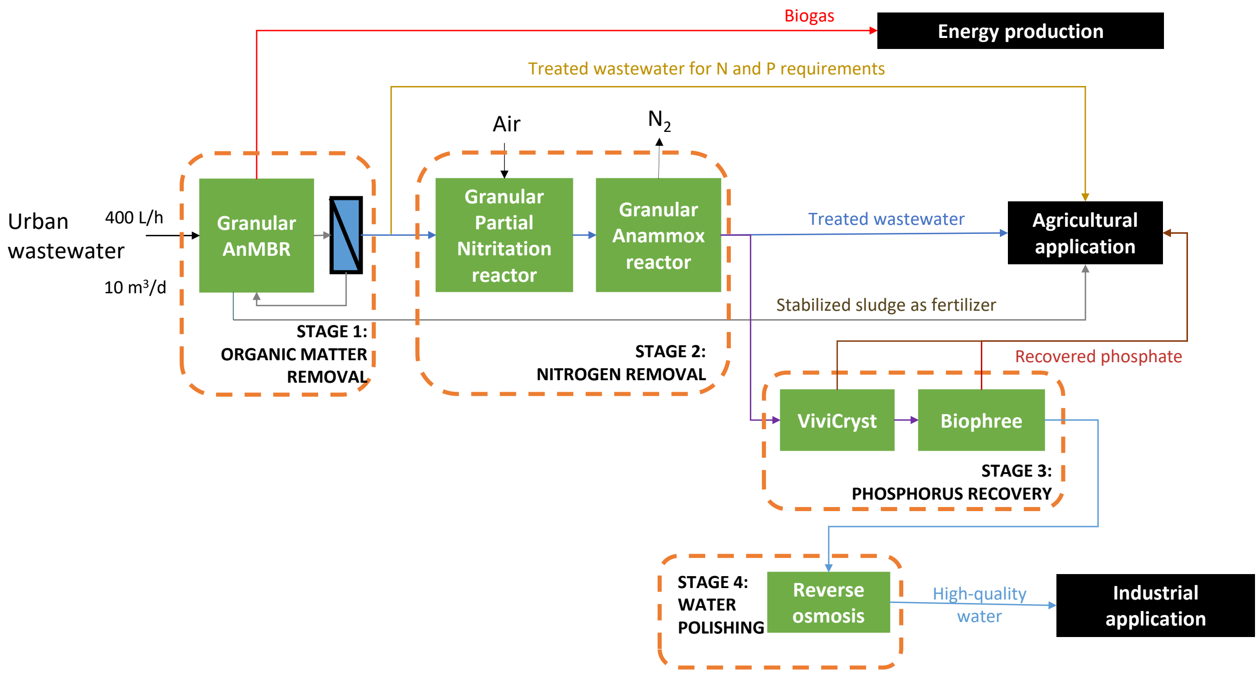

Figure 2. Process diagram for CS5

2. Design parameters for CE processes: CS4

2.1. Relevant results from bench scale tests for the design of the pilot units for CE processes

The CS4 aims to demonstrate the feasibility of implementing technologies to treat the produced effluent from the wastewater treatment plant (WWTP) of Larnaca (Cyprus). Currently, the salinity of the produced effluent is 1.5 g L^{-1}. This stream with this salinity level cannot be used for irrigation of sensitive cultivations, while long-term use of this water for irrigation can lead to salt accumulation in soil.

Furthermore, water produced by Larnaca WWTP has a phosphorus (P) content of 0.5 mg L^{-1}, which can stimulate algae development in the lagoons where the effluent stays before passing through the chlorination unit to the irrigation water supply system. Algae can cause problems to the water irrigation pipes or other watering system components.

As a solution to those issues arising by using the effluent from the WWTP, a pilot system will be installed and operated in Larnaca WWTP. The first stage of the process is phosphate recovery with the BioPhree technology. The P concentration in the effluent will be decreased by BioPhree from 0.5 mg L^{-1} to 10-40 ppb. At these low levels biological growth is limited. This step will also protect the membranes of the next systems from biofouling. After the BioPhree, a Nanofiltration (NF) unit is applied to remove calcium and magnesium ions, from the effluent. Concentrate from the NF passes through a low-temperature evaporator (LTE), removing divalent ions to provide low salinity water. The NF permeate passes through a reverse osmosis (RO) system, which produces a high-quality permeate with <100 µS cm^{-1} conductivity. The RO concentrate will pass through a multi-effect distillation (MED) evaporator: The MED evaporator will concentrate the brine from RO, also producing distillate water (condensate). Finally, the brine concentrate from the evaporator will pass through the crystallizer. In the crystallizer, the brine will be further concentrated. The products of the crystallizer will be distillate water (condensate), and a saturated solution which can be used in the WWTP chlorination unit

During the project's first months, bench-scale tests were conducted by NTUA, previous to the construction of this pilot system to obtain design and operational parameters for the Nanofiltration Unit and the Reverse Osmosis Unit.

2.1.1. Membrane Bioreactor Effluent from WWTP of Larnaca.

At the first two months of the project, samples from the effluent of MBR were taken and analyzed by Larnaca, NTUA, and external laboratories. In the following table (Table 1) a typical composition of the MBR Effluent is presented. As it is shown, the total phosphorus is 1.52 mg L^{-1} aiming to be removed by the Biophree technology constructed by Wetsus since it could potentially lead to algae development in the lagoons. The high amount of TSS could be a problem for the NF unit since could potentially block the membranes. The WWTP is aware of this high value and is addresing this issue.

Table 1. Typical composition of MBR effluent of Larnaca WWTP

Element	Symbol	Value	Unit
Sodium	Na	490	mg L^{-1}
Potassium	K	43	mg L^{-1}
Magnesium	Mg	61	mg L^{-1}
Calcium	Ca	149	mg L^{-1}
Zinc	Zn	42	μg L^{-1}
Copper	Cu	14	μg L^{-1}
Lead	Pb	<75	μg L^{-1}
Cadmium	Cd	<0.5	μg L^{-1}
Mercury	Hg	<0.4	μg L^{-1}
Chromium	Cr	6.6	μg L^{-1}
Nickel	Ni	8.1	μg L^{-1}
Boron	B	0.67	mg L^{-1}
Chloride	Cl	890	mg L^{-1}
Tot. Phosphorous	P	1.52	mg L^{-1}
Tot. Nitrogen	N	10.4	mg L^{-1}
Elec. conductivity	EC	4.2	mS cm^{-1}
pH	pH	7.9	
BOD_5	BOD_5	<1	mg L^{-1}
COD	COD	20	mg L^{-1}

Tot. Susp. Solids	TSS	7	mg L^{-1}

As can be concluded from Table 1, the conductivity of treated wastewater is mainly due to Na^+ and Cl^- ions (effluent contains about 1.5 g L^{-1} sodium chloride).

2.1.2. Bench-scale tests with the NF and RO units.

Bench-scale tests were conducted by the NTUA team using NF and RO units (single module) with a capacity of about 0.5 m^3 h^{-1} and synthetic solutions.

The synthetic solution was prepared in the laboratory of NTUA to simulate the real effluent of MBR with the major ions of this effluent (Table 2).

Table 2. The concentrations of major ions in the brine used for the bench-scale

Element	Symbol	Value	Unit
Sodium	Na	559	mg L^{-1}
Potassium	K	2.5	mg L^{-1}
Magnesium	Mg	36	mg L^{-1}
Calcium	Ca	228	mg L^{-1}
Chloride	Cl	875	mg L^{-1}
Sulfates	SO4	47	mg L^{-1}

The objectives of the bench-scale tests conducted in the Lab of Environmental Science and Technology Unit of NTUA were to find:

- the highest recovery of NF and RO membranes
- the efficiency of NF for separating monovalent ions from multivalent
- the quality of permeate and the characteristics of concentrate streams of these membranes

2.1.2.1. Nanofiltration Unit – Bench-scale tests

The NF unit aims to separate the divalent ions (Ca^{+2}, Mg^{+2}, $SO4^{-2}$) from the monovalent ions (Na^+, Cl^+, K^-) of the inlet stream, to produce a high-purity NaCl solution for the RO unit. In this way, the membranes of the RO unit are also protected from scaling.

The membrane used in the NF unit was a FILMTEC NF270-4040 membrane which is a Polypiperazine Thin-Film Composite membrane, suitable for TOC and medium hardness removal. The FILMTEC

NF270-4040 (4'' module) has an active area of 7.6 m^2 and can achieve 97% salt rejection in medium hardness influent. In the following table (Table 3) the main operating limits of the membrane are presented.

Table 3. Main features of the NF270-4040 membrane

Feature	Value
Maximum Operating Temperature	45°C
Maximum Operating Pressure	41 bar
Maximum Feed Flow Rate	3.6 m^3 h^{-1}
Maximum Pressure Drop	0.9 bar

As it is shown inFigure 3, the NF system consists of a feed tank, a cartridge filter, a high-pressure pump, and an NF270-4040 membrane. The feed pump circulates the synthetic solution from the inflow tank to the cartridge filter (pre-treatment stage). That filter has a standard 5-micron pore size and protects the main membrane. Then, a high-pressure pump leads the brine into the NF270-4040 where the separation of the ions is performed. To this pilot system, the operator can adjust the retentate and permeate flow and at the same time recirculate the retentate into the membrane. The recirculation of the retentate is the most used technique to protect membranes from operational problems such as scaling.

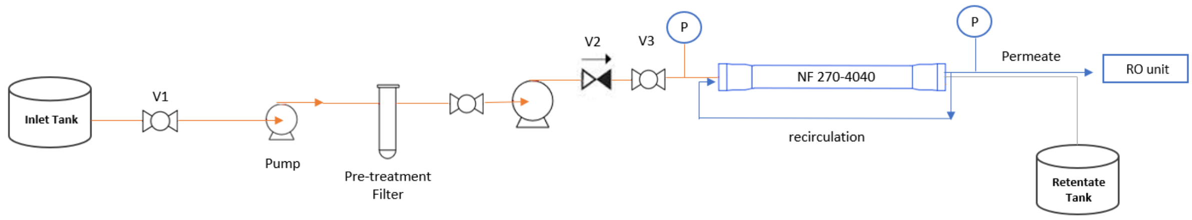

Figure 3. P&ID of the NF unit

The objectives of the bench-scale tests were to find out:

- The highest possible divalent ions rejection.
- The lowest monovalent ions rejection.
- The highest permeate recovery.

The NF unit was operated in a batch mode and the synthetic solution prepared in the laboratory of NTUA was used as an influent. Three groups of experiments (Table 4) with different recoveries and recirculations were performed. Furthermore, the addition of hydrochloric acid solution for pH correction from 7.3 to 6.0 was tried. The decrease of inflow pH was suggested to minimize scaling on the membranes as well as the need for antiscalants use.

Table 4. The 5 groups of experiments that were held with the NF unit

	Membrane Recovery	Recirculation	pH
1st group of experiments	75%	67%-75%-80%-83%	7.3
2nd group of experiments	60%	56%-65%-71%-76%	7.3
3rd group of experiments	50% - 60%-70%	Without recirculation	7.3
4th group of experiments	50%-60%-75%	Without recirculation	6
5th group of experiments	75%	Without recirculation	6

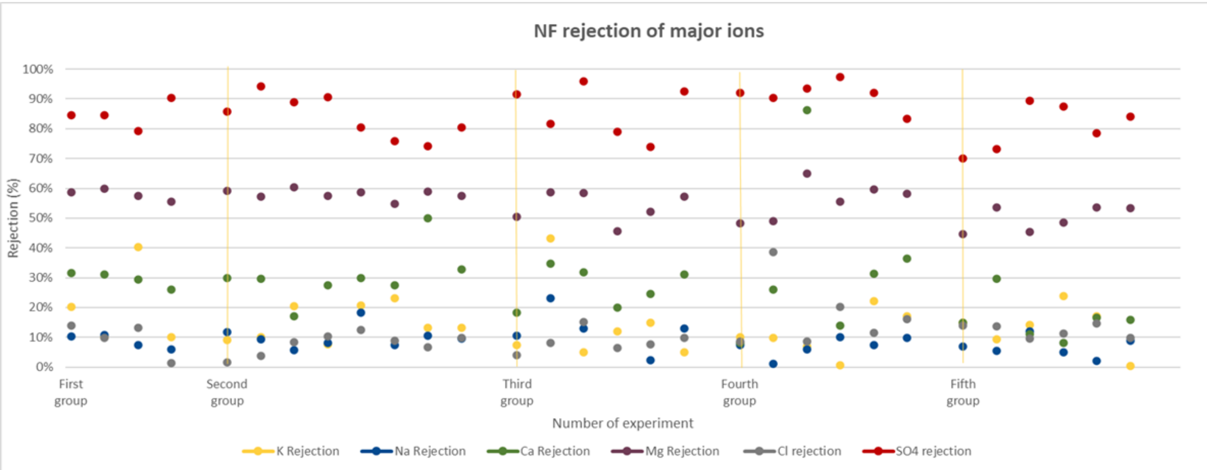

Figure 4. NF rejections of major ions

As it can be seen in Figure 4, the NF 270-4040 membrane exhibits low monovalent ions rejection (about 10%), medium divalent cations rejections (Mg^{2+} about 60 %, and Ca^{2+} rejection about 30%), and very high sulfate (SO_4^{2-} anions) rejection, which reaches 96%. The rejection efficiencies of all considered ions seem almost independent from concentrate recirculation flow.

As it is not possible to buy install and test many different membranes on the NF system, the WAVE, DuPont Design Software, was used to investigate the effectiveness and rejection factors of different types of membranes. To ensure the reliability of this software in the case of our brine, we examine the rejection factors and the effectiveness of membrane NF270- 4040. The rejection factors from the WAVE were similar to those experimentally observed. At the following figure (Figure 5) the comparison of the rejections factors of the main ions are presented.

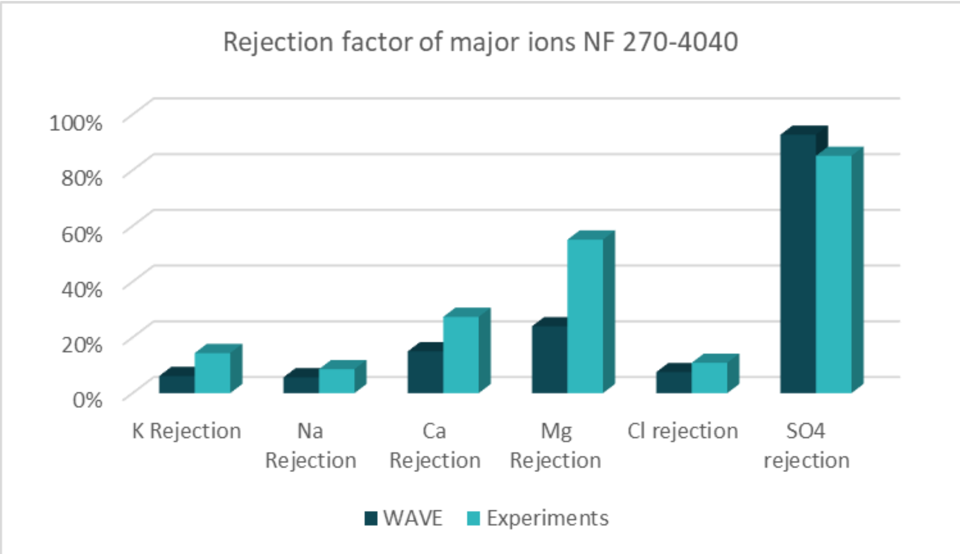

Figure 5. Comparison of rejection factors from WAVE and from the Experiments

At the following tables (Table 5, Table 6, Table 7) the main values and characteristics of the membrane are presented. The system that was simulated consisted of 6 elements (membranes) NF 270-4040 and the system recovery was set at 75%. During the process the pressures for the feed, concentrate and permeate were 1.7, 1.0, 0 respectively. The feed TDS was 2,547 mg L^{-1}, the concentrate 2,874 mg L^{-1} and for the permeate 1,929 mg L^{-1}. The pH was adjusted on WAVE at 6 to avoid scaling thus no design or scaling issues were occurred.

Table 5. WAVE results from NF 270-4040: operational conditions

Pass	NF 270-4040
Stream name	Larnaca wastewater
Water type	Well Water (SDI<3)
Number of elements	6
Total active area (m^2)	45.7
Feed flow per pass (m^3 h^{-1})	1.30

Feed TDS (mg L^{-1})	2,547
Feed Pressure (bar)	2
Flow Factor Per Stage	0.85
Permeate Flow per Pass (m^3 h^{-1})	0.45
Pass Average flux (LMH)	9.9
Permeate TDS (mg L^{-1})	1,929
Pass recovery	34.6%
Average NDP (bar)	0.8
Specific Energy (KWh m^{-3})	0.20
Temperature (°C)	23
pH	6
NF System Recovery	75%

Table 6. WAVE results from NF 270-4040: effluents characteristics

Stage	Elements	Feed			Concentrate			Permeate			
		Feed Flow (m^3 h^{-1})	Recirc. Flow (m^3 h^{-1})	Feed Pressure (bar)	Conc Flow (m^3 h^{-1})	Conc Pressure (bar)	Press Drop (bar)	Perm Flow (m^3 h^{-1})	Avg Flux (LMH)	Perm Press (bar)	Perm TDS (mg L^{-1})
1	NF270-4040	1.30	0.70	1.7	0.85	1.0	0.7	0.45	9.9	0.0	1,929

Table 7. WAVE results from NF 270-4040: ions concentrations

Sodium	Symbol	Feed	Concentrate	Permeate
		mg L^{-1}	mg L^{-1}	mg L^{-1}
Potassium	K^-	1.3	1.53	1.22
Sodium	Na^+	500.0	585.5	471.6
Magnesium	Mg^{-2}	41.0	71.2	30.96

Calcium	Ca^{-2}	240.0	351.0	203.1
Chloride	Cl^{-}	1,220.0	1,494.0	1,128.0
Sulfates	SO_4^{-2}	50.0	189.5	3.62

Some more membranes such as NF200-4040 and NF90-4040 were tested through this software. The Membrane NF90-4040 resulted in the highest rejections factors among the tested membranes, namely about 90% for Ca^{+2} and Mg^{+2}, 96% for SO_4^{-2}, and 75% for Na^{+} and Cl^{-}.

At the following tables (Table 8, Table 9, Table 10) the main values and characteristics of the membrane NF 90-4040 are presented. The system that was simulated consisted of 6 elements and the system recovery was set at 71.5%. During the process the pressures for the feed, concentrate and permeate were 7.5, 6.0, 0 respectively. The feed TDS was 4,840 mg/L, the concentrate 7,684 mg/L and for the permeate 420.5 mg/L. The pH was adjusted on WAVE at 5 to avoid scaling thus no design or scaling issues were occurred.

Table 8. WAVE results from NF 90-4040: operational conditions

Pass	NF 90-4040
Stream name	Larnaca wastewater
Water type	Well Water (SDI<3)
Number of elements	6
Total active area (m^2)	45.7
Feed flow per pass ($m^3\ h^{-1}$)	2.31
Feed TDS (mg L^{-1})	4,840
Feed Pressure (bar)	7.8
Flow Factor Per Stage	0.85
Permeate Flow per Pass ($m^3\ h^{-1}$)	0.91
Pass Average flux (LMH)	19.8
Permeate TDS (mg L^{-1})	420.5
Pass recovery	39.4%
Average NDP (bar)	2.7

Specific Energy (KWh m^{-3})	0.69
Temperature (°C)	25
pH	5
NF System Recovery	71.5%

Table 9. WAVE results from NF 90-4040: effluents characteristics

Stage	Elements	Feed			Concentrate			Permeate			
		Feed Flow (m^3 h^{-1})	Recirc. Flow (m^3 h$^-$)	Feed Pressure (bar)	Conc Flow (m^3 h$^-$)	Conc Pressure (bar)	Press Drop (bar)	Perm Flow (m^3 h$^-$)	Avg Flux (LMH)	Perm Press (bar)	Perm TDS (mg L^{-1})
1	NF90-4040	2.31	1.13	7.5	1.41	6.0	1.5	0.91	19.8	0.0	420.5

Table 10. WAVE results from NF 90-4040: ions concentrations

Sodium	Symbol	Feed	Concentrate	Permeate
		mg L^{-1}	mg L^{-1}	mg L^{-1}
Potassium	K^-	1.31	4.41	0.34
Sodium	Na^+	502.2	1,712	126.2
Magnesium	Mg^{-2}	41.18	158.7	4.68
Calcium	Ca^{-2}	241.1	930.1	26.99
Chloride	Cl^-	1,275	4,565	252.6
Sulfates	SO_4^{-2}	49.78	203.1	2.14

The FilmTec™ NF90-4040 Membrane Elements are Polyamide Thin-Film Composite membrane type which provides high productivity performance with the highest salt and chemical contaminants rejection, low energy consumption, and good fouling resistance. In Table 11, the main operating limits of the membrane are presented and as it can be seen they are similar to the NF270-4040 that was experimentally tested.

Table 11. Main features of the NF90-4040 membrane

Feature	Value
Maximum Operating Temperature	45°C
Maximum Operating Pressure	41 bar
Maximum Feed Flow Rate	3.6 m^3 h^{-1}
Maximum Pressure Drop	0.9 bar

2.1.2.2. RO unit – Bench Scale tests

The RO unit aims to remove most of the remaining ions from the NF permeate. The influent contains mainly monovalent ions (Na^+ and Cl^-) and during the operation, the separation of the water and those ions is achieved.

The RO unit that was used for the bench-scale tests is a single module unit of 0.5 m^3 h^{-1} capacity. The membrane that is installed in the system is the XLE-2540. XLE membranes are usually used in RO technologies since they provide consistent and reliable system performance. The XLE-2540 has the following operating limits (Table 12) and has been used for the bench-scale tests aiming to achieve:

- The highest permeate recovery (i.e the lowest volume of retentate at the end of the process).
- High purity water

Table 12. Main features of the XLE-2540 membrane

Feature	Value
Maximum Operating Temperature	45°C
Maximum Operating Pressure	41 bar
Maximum Feed Flow Rate	3.2 m^3 day^{-1}
Maximum Pressure Drop	0.9 bar

The RO system consists of a feed pump that circulates the inflow (in our case the NF permeate) from the inlet tank to the cartridge filter (pre-treatment stage) that has a standard 5-micron pore size and protects the main membrane. Then, a high-pressure pump passes the brine into the XLE-2540 membrane. The permeate, concentrate, and recirculation flow can be adjusted manually (Figure 6).

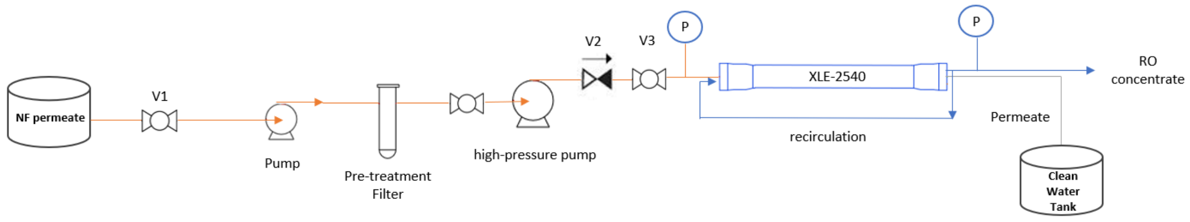

Figure 6. P&ID of the RO unit

For the RO unit experiments, the NF permeate from the final set of experiments was driven to the RO unit, and new concentrate and permeate streams are produced. The RO unit was set to work at the highest possible recovery, 90%. The concentration of retentate at the end of the process ranged from 7 g L^{-1} to 14 g L^{-1} while permeate was low salinity water appropriate for irrigation use after alkalinity recovery. The system achieved high rejection factors, more than 90% for divalent ions, although there was some small passage of monovalent cations (Na^+, K^+) (Figure 7).

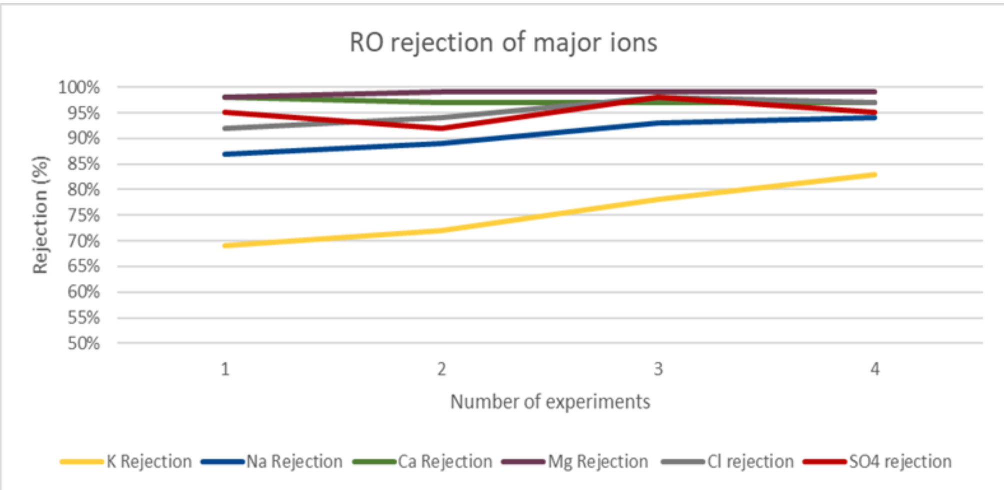

Figure 7. RO rejection of major ions

Membrane FILMTECH LC HR 4040 was also tested through DuPont WAVE software. The rejection factors for FILMTECH LC HR 4040 were higher than 98% for all ions.

As it can be seen from the following Figure 8, the LC HR 4040 membrane had similar rejections factor than the experiments performed with the pilot system.

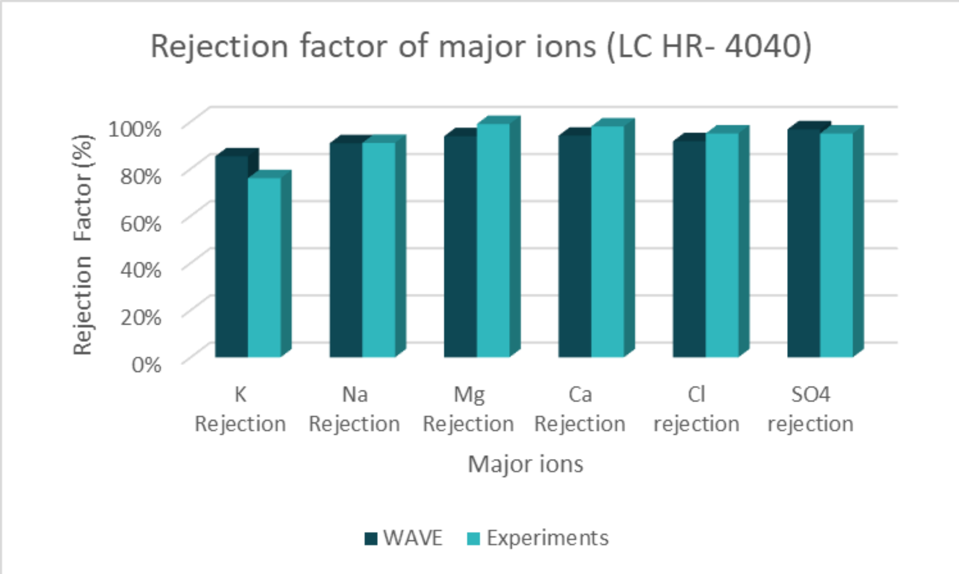

Figure 8. LC-HR 4040 ion rejection in comparison to actual experiments

At the following tables (Table 13, Table 14,Table 15) the main values and characteristics of the membrane are presented. The system that was simulated consisted of 4 elements (membranes) LC-HR 4040 and the system recovery was set at 71.5%. During the process, the pressures for the feed, concentrate and permeate were 9.5, 9.1, 0 respectively. The feed TDS was 2,487 mg L^{-1}, the concentrate 6,163 mg L^{-1} and for the permeate 37.91 mg L^{-1}. The pH was adjusted on WAVE at 5 to avoid scaling.

Table 13. WAVE results from LC HR 4040: operational conditions

Pass	LC HR-4040
Water type	Wastewater (With conventional pretreatment, SDI<5)
Number of elements	4
Total active area (m^2)	34.9
Feed flow per pass (m^3 h^{-1})	1.42
Feed TDS (mg L^{-1})	2,487
Feed Pressure (bar)	9.8
Flow Factor Per Stage	1

Permeate Flow per Pass ($m^3\ h^{-1}$)	0.85
Pass Average flux (LMX)	24.3
Permeate TDS (mg L^{-1})	37.91
Pass recovery	59.9%
Average NDP (bar)	6.1
Specific Energy (KWh m^{-3})	0.56
Temperature (°C)	25
pH	4.5
NF System Recovery	71.5%

Table 14. WAVE results from LC HR 4040: effluents characteristics

Stage	Elements	Feed			Concentrate			Permeate			
		Feed Flow ($m^3\ h^{-1}$)	Recirc. Flow ($m^3\ h^{-1}$)	Feed Pressure (bar)	Conc Flow ($m^3\ h^{-1}$)	Conc Pressure (bar)	Press Drop (bar)	Perm Flow ($m^3\ h^{-1}$)	Avg Flux (LMH)	Perm Press (bar)	Perm TDS (mg L^{-1})
1	LC HR-4040	1.42	0.51	9.5	0.57	9.1	1.5	0.85	24.3	0.0	37.91

Table 15. WAVE results from LC HR 4040: ions concentrations

Sodium	Symbol	Feed	Concentrate	Permeate
Unit		mg L^{-1}	mg L^{-1}	mg L^{-1}
Potassium	K^-	0.34	4.76	0.05
Sodium	Na^+	126.2	1,846	11.54
Magnesium	Mg^{-2}	4.68	70.61	0.29
Calcium	Ca^{-2}	26.98	407.7	1.60
Chloride	Cl^-	252.6	3,727	20.96

Sulfates	SO_4^{-2}	2.14	33.18	0.07

Type LC HR 4040, which gives the highest rejection factors for all ions, was selected to be installed in the pilot system.

2.1.3. BioPhree technology

For BioPhree no bench-scale tests have been performed in the Water Mining project, as this technology was already tested at pilot scale. In 2018, BioPhree was demonstrated at pilot scale in the George Barley Water Prize (GBWP) contest for 3 months in Ontario, Canada. Results from that period have been taken into account in the design of the BioPhree pilot installation for CS4. The relevant results from the GBWP that aided in the design of the pilot unit are described here.

As the pilot in the GBWP was demonstrated on surface water in the Holland Marsh, there are differences to take into consideration when designing a pilot installation for WWTP effluent.

The influent of the BioPhree pilot in the GBWP had a phosphorus content of about 0.3-0.4 mg L^{-1} P. On a yearly average, the total phosphorus content of the permeate from WWTP Larnaca was 1.6 mg L^{-1} as taken from measurement data supplied by LSDB.

An important parameter in the design of an adsorption process is the empty bed contact time (EBCT). This is the time that the water to be treated is in contact with the adsorption material bed in the column. Sufficient time is needed for P to attach to the adsorption material. The GBWP pilot operated with varying flowrates. With a low flowrate of 9.5 m^3 per day, the EBCT was 56 minutes, and effluent concentrations of 15-16 µg L^{-1} were reached. With a higher flowrate of 31 m^3 per day, the EBCT was 18 minutes and an effluent concentration of 40 µg L^{-1} was reached. The effluent concentration target for Larnaca is to always be below 50 µg L^{-1}.

An important difference between the case study sites and Canada is the temperature. Earlier lab tests showed that adsorption kinetics, and adsorption capacity of the adsorbent increase with temperature. As the BioPhree pilot will be demonstrated in Larnaca and Barcelona, a lower EBCT can be allowed. For an influent flow of 1 m^3 h^{-1}, the adsorption bed has been dimensioned to have an EBCT of 14 minutes. This parameter is adjustable by filling the adsorption column with more or less adsorption material. For a flow of 1 $m^3 h^{-1}$ an EBCT in the range 5 to 20 minutes can be used with the current design.

The adsorption columns are extremely flexible to varying ortophosphate (oP) concentrations. The current EBCT times are based on experiences in the lab and pilot stage with significant higher oP loads (up to 5-10 times higher). Therefore, the design will be able to also handle higher oP loads then are now anticipated. The main effect of higher loads will be a quicker saturation of the adsorption columns, thus increasing demands for the regeneration of the adsorbent. The need for regeneration can be easily anticipated by monitoring the phosphate concentrations of the influent and effluent of the columns. In the pilot design an online phosphate analyzer has been implemented for this purpose.

The number of regenerations has also been considered in the pilot design. A research objective of Wetsus is to gain better understanding of how many regenerations affect the quality and performance of the adsorbent. Therefore, it is necessary to perform a high number of regenerations per year. The design consideration was to have a large enough bed volume to provide an adequate EBCT but have the bed as small as possible to have sufficient regenerations.

A bed volume of 150L, resulting in 14 minutes EBCT and approximately 10 regenerations per year per column was chosen as a final design parameter for CS4.

Another important difference between treating water from a lake and permeate from a WWTP is the amount of suspended particles. The BioPhree pilot demonstrated in the GBWP had three filtration units to remove all particulates to the range of 0 to 0.5 ppm TSS. In the design for CS4 this has been adjusted to one unit.

2.2. Definition of the design parameters of the pilot units for CE processes

2.2.1. NF and RO units

The design of the NF and RO pilot units was based on predefined feed, permeate and concentrate flow rates, as well as a predefined feed water analysis.

The target for NF and RO units was to achieve the highest possible permeate recoveries with adequate salt rejections. During bench-scale tests, as already shown, several commercial NF membranes were tested experimentally and through the WAVE software tool. The composition of permeates and concentrates of these membranes was determined. This way, the most suitable NF and RO membranes that separate the monovalent ions from multivalent ions were found and selected to be used in the pilot unit. The NF90-4040 and the LC HR 4040 membranes were selected for the NF and RO units, respectively since those membranes meet the high rejection and recovery requirements. Also, the use of chemical antiscalants, including dosing rate and pH adjustments are taken into consideration since they affect the overall operation of the systems.

The system parameters such as permeate and concentrate flow, percentage of recirculation, membrane flux, operating pressure, pH, and composition of resulting streams were estimated and verified using calculation tools to be sure that all values are within acceptable operating ranges without design warnings. Automated system operation is allowed by the selection of appropriate equipment, instrumentation, and control system.

A single pass NF and a double pass RO system have been designed considering the feedwater composition and flow rate, the specific membrane characteristics, and recovery. The P&ID diagram and 3D design of the NF and RO unit including all required equipment and instruments were drawn to facilitate the construction and assembly of the pilot unit. The resulted PFD of the NF pilot unit is given in Figure 9.

The MED and LTE evaporator as well as the Crystallizer (the rest three units of the proposed pilot system) are already constructed and tested in previous projects. They will operate for the CS4 aiming to recover clean water, NaCl, magnesium, and calcium salts.

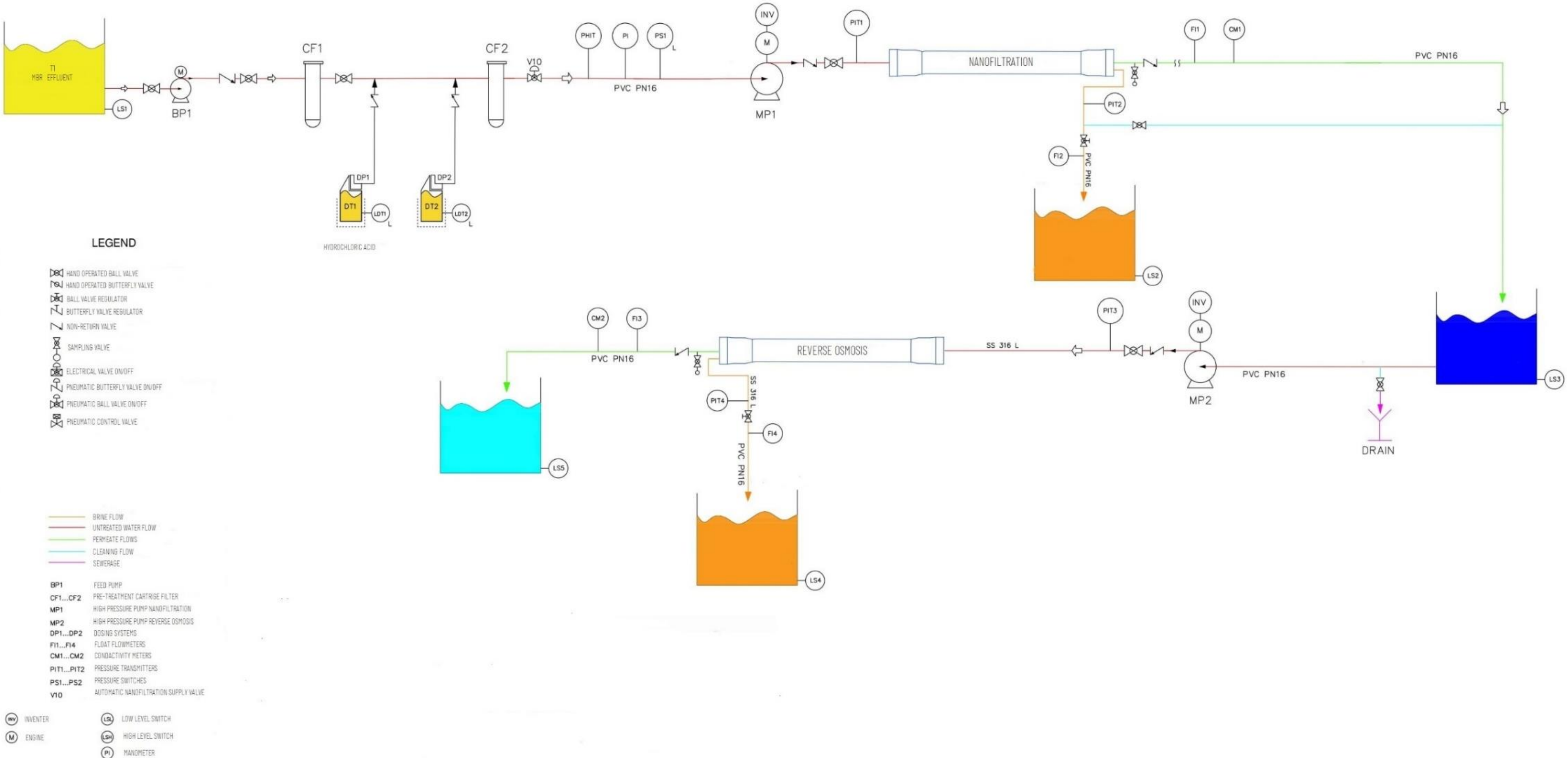

Figure 9. P&ID of the NF and RO pilot units

2.2.1. Biophree unit

A Process flow diagram of the BioPhree unit is shown in Figure 10.

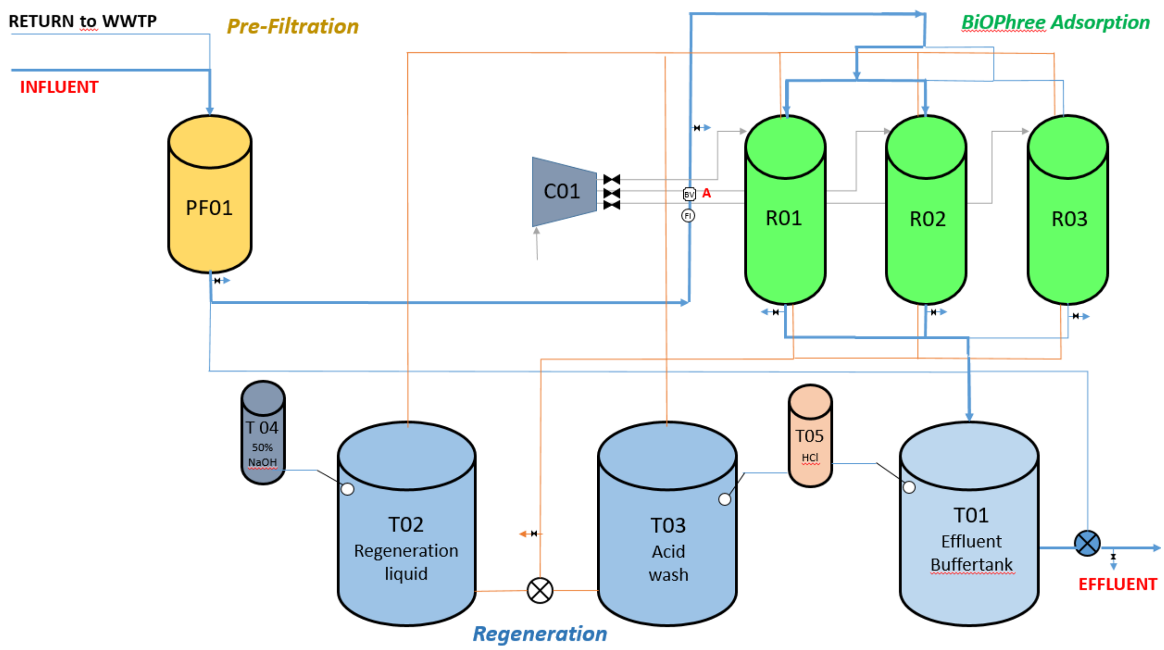

Figure 10. Process Flow Diagram of the BioPhree pilot installation

A short description of the most important design parameters is given below per unit operation:

Influent: Can be pumped in by an influent pump or can be received pressurized in which case the influent pump is bypassed. Influent flow is controlled automatically by flowmeters and needle valves to 1.0 $m^3 h^{-1}$ for CS4 and 0.4 L h^{-1} in CS5

Filtration: Filtration column (PF01) filled with activated filter media (AFM). Automatic monitoring of pressure drop and backwash if pressure drop becomes too high due to filter clogging. The column has an area of 0.20 m^2 and a total volume of 175 L.

Adsorption: Three 250L columns in parallel. Always two in operation and one in standby or being regenerated. Filled with an adsorbent volume of 150L each to provide an EBCT of 14 minutes. The adsorbent used is iron-hydroxide based granules. Expected regenerations are at least 10 per year per column. Flow is controlled to 0.5 $m^3 h^{-1}$ over each column.

P-Analyzer: Automatic monitoring of P concentrations in influent and effluent of each column.

Effluent: Buffer tank for effluent. If pH is higher than 8.0 after regeneration it is corrected automatically by HCl dosing to pH lower than or equal to 8.0.

Regeneration liquid: The pH is constantly measured and OH concentration adjusted after each regeneration to correct pH by dosing of concentrated NaOH.

Acid wash: pH controlled by dosing of concentrated HCl.

Compressor: A compressor (C01) is used to push out remaining liquid of the columns when switching between operation and regeneration.

2.3. Pilot construction and next steps

The bench scale tests results suggested that the NF unit is appropriate for the separation of Mg^{+2} and Ca^{+2} and RO unit for the separation of NaCl, from the WWTP effluent. The two concentrated streams produced by these two units can be further processed by the LTE and MED evaporator-Crystallizer, to recover valuable salts and minerals.

Details about the operation, capacity and main parameters of MED and crystallizer are mentioned in the Deliverable 5.1 "Bench-scale tests for CS4 & CS5".

The NF and RO pilot units were assembled in NTUA facilities. NF unit consists of two pressure vessels with three membrane elements each vessel (NF90-4040) and the RO unit consists of two pressure vessels with two membrane elements each (LC HR 4040). The capacity of the two systems is 1 m^3 h^{-1} and the continuous operation is monitored and controlled remotely via PLC system.

The units were mounted in a 20 ft container and transferred to Cyprus. In Figures 11 – 13, photos from the installation of the containers and their interior are presented. The commissioning and the start-up of the unit conducted in January 2022. The plant commissioning and operation is carried out by qualified personnel.

In case of the BioPhree unit, the complete installation was built in a 40-foot shipping container and shipped to Larnaca (Figure 14).

Figure 11. The NF, RO, MED, and Crystallizer installation in Larnaca

Figure 12. MED and Crystallizer Units

Figure 13. The NF and RO units

Figure 14. BioPhree unit in Larnaca

water mining

3. Design parameters for CE processes: CS5

3.1. Relevant results from bench scale tests for the design of the pilot units for CE processes

3.1.1. Granular AnMBR reactor

Bench-scale tests related to subtask 5.1.1 allowed to define the best technology to implement at pilot scale in CS5 for the granular AnMBR. Detailed information can be found on Deliverable D5.1. Briefly, the performance of an Upflow Anaerobic Sludge Blanket (UASB) reactor was compared with an Expanded Granular Sludgle Blanket (EGSB) reactor, both connected to an ultrafiltration unit, to achieve anaerobic digestion at mainstream conditions (15 ºC and ≈300 mg L^{-1} of COD). Both systems achieved proper performance at the studied conditions: ≈77 % of COD removal, < 60 mg L^{-1} COD in the permeate and flux of 12-33 LMH. However, higher organic loading rate (OLR) was achieved with the EGSB reactor in comparison to the UASB reactor (3.9±1.3 kg COD m^{-3} d^{-1}), while permeate quality and removal efficiencies were similar. The maximum OLR achieved by the granular AnMBR was 19.0±7.0 kg COD m^{-3} d^{-1}. This fact can be mainly related to the applied upflow velocities (Vs). UASB reactor worked until 3.9±1.3 m h^{-1}, however EGSB reactor achieved good performance at 6.2±0.2 m h^{-1}. However, due to the limited time to define the applied OLR in the pilot plant and perform the preliminary designs for the selection of the engineering company, a OLR of 10.7 kg COD m^{-3} d^{-1} was selected.

This information was used for the design of the prototype for the CS5, mainly for sizing the reactor and determine the UF conditions.

3.1.2. Partial nitritation reactor

Subtask 5.1.1 allowed to determine the conditions to perform partial nitritation (PN) at mainstream conditions. Detailed information can be found on Deliverable D5.1. Main information is related to the operational conditions for the start-up of the partial nitritation reactor. As aerobic granular sludge enriched with ammonium-oxidizing bacteria (AOB) is not widely available at pilot or full scale, it is necessary to form granule from flocs, as they are conventionally found in urban WWTP. It was investigated a start-up strategy to form granules performing partial nitritation: to feed a wastewater rich in ammonium (reject wastewater from an urban WWTP) at mesophilic conditions (32-35 ºC) and operating the reactor as a sequencing batch reactor (SBR). The start-up strategy consisted in promoting high ammonium oxidation to nitrite, while limiting the nitrate production, due to high ammonium concentration inside the reactor and low dissolved oxygen (DO) concentration, to promote repression of nitrite oxidizing bacteria (NOB) and to allow the growth of AOB at high nitrogen loading rate (NLR) (0.8-1.2 kg N m^{-3} d^{-1}). Thus, this strategy will be implemented at pilot scale, to produce granular biomass.

When partial nitritation was studied at mainstream conditions and continuous operation was stablished, nitrate build-up in the system occurred. This fact could be mainly related to the low biomass concentration inside the reactor (0.5 g VSS L^{-1}). Thus, microbial activity was limited and strongly affected by the low temperature (15 ºC) and low ammonium concentration (< 25 mg N L^{-1}). Under these conditions, NOB could growth and nitrite was oxidized to nitrate. Considering the performance of the bench-scale reactor, a conservative value of the nitrogen loading rate (0.5 kg N m^{-3} d^{-1}) was used for the design of the PN reactor. To further demonstrate the partial nitritation at mainstream conditions, avoiding nitrate build-up, different NOB inhibition strategies could be implemented at pilot scale, such as (i) working at very low DO concentrations (< 0.5 mg L^{-1}), (ii) performing NOB inhibition by free nitrous acid instead of ammonia and (iii) enhance biomass retention by applying inert support to develop biofilm.

3.1.3. Anammox reactor

Anammox process, at mainstream conditions, was able to be operated at a NLR 0.2 kg N m^{-3} d^{-1}, but low nitrogen removal (< 60 %) and high concentration of nitrogen in the effluent (21 mg N L^{-1}) was achieved. Anammox process could be limited due to the nitrate formation in the feeding tank due to the oxidation of nitrite, the low nitrite to ammonium ratio applied and the low biomass concentration and/or anammox activity. Detailed information can be found on Deliverable D5.1, related to subtask 5.1.1.

As it is expected that better performance of the anammox reactor could be achieved, the pilot plant was designed to apply an NLR of 0.5 kg N m^{-3} d^{-1}. This value is related to the fact that UASB reactors performing partial nitritation can achieve NLR higher than 2 kg N m^{-3} d^{-1} working at mainstream conditions and high nitrogen removal (> 75 %) (Ma et al., 2013; Reino et al., 2018).

3.1.4. ViviCryst technology

After deliberating with Wetsus' partner Royal Haskoning DHV, some new insights about the crystallization process were gained, which resulted in relevant modifications of the design of the crystallization unit. Most importantly, it seems that the previous design tried to combine two methods of crystallization: Crystallization on the sand, and crystal growth of particles. By re-introducing the fines into the crystallizer, they actually 'compete' with the sand and influence the crystallization.

Furthermore, the importance of 'loading' was discovered. In the performed experiments with low influent P concentration, the low loading of 0.1 kg P m^{-2} h^{-1} could be the cause why vivianite is being observed. Therefore, Wetsus has redesigned the bench-scale reactor and performed further tests.

The previous design is presented in Figure 15. The crystallizer was composed of a vertical column - where the reaction takes place -, a settler - where all the particles eluded from the main column can be gathered and collected -, and three different pumps, used for P dosing, Fe dosing and the reflux pump to control the up-flow velocity. The main column of the crystallizer is composed of three different parts that are connected to a sudden enlarged joint. The diameter of each part increases as we move from the bottom to the top. With this expansion of the diameter in each part, the up-flow velocity decreases from 50 cm min^{-1} to 19 cm min^{-1} to 8 cm min^{-1} for section 1, 2 and 3 respectively. This traps the fines and promotes crystal agglomeration. Additionally, at the lowest part of the main column 315 g of sand was placed (approximate height: 15 cm) to create a "sand bed" or "vivianite seed bed", where all the particles can stick together and form the crystals.

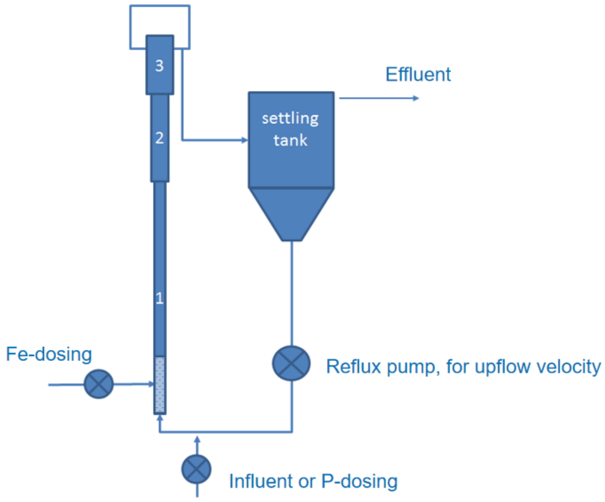

Figure 15. Diagram of the previous bench-scale crystallizer

The redesigned crystallizer is pictured in Figure 16. Fines are no longer recirculated. In fact, recirculation has been removed entirely. The upflow velocity now is fully provided by the influent flow.

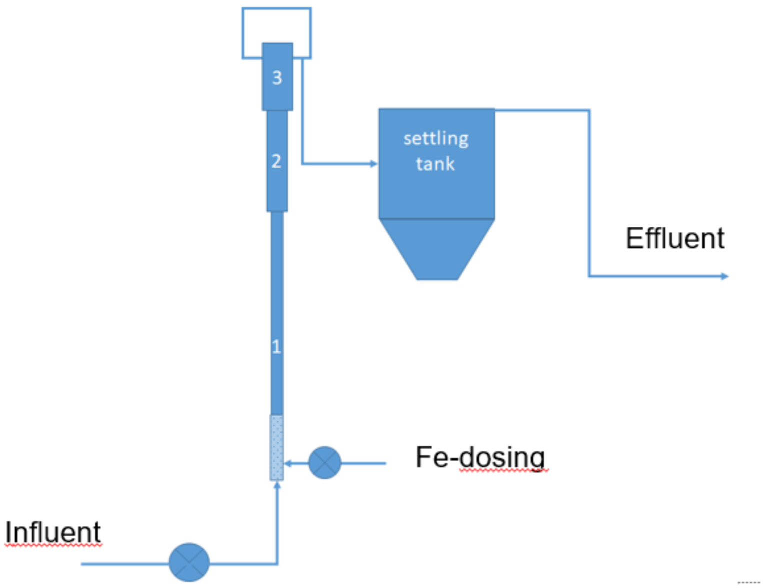

Figure 16. Adjusted setup of the bench-scale crystallizer

The first test with the new bench-scale design were performed at following conditions:

- Influent phosphorus concentration: 20 mg L^{-1}
- P loading: 0.6 kgP m^{-2} h^{-1}

- Fe/P molar ratio: 2.5 (54 mg L^{-1} Fe)
- Overdosing: 0.65 mmol/L (36 mg L^{-1} Fe)
- Upflow velocity: 50 cm min^{-1} or 30 m h^{-1}
- Sand bed: 300g. 15 cm height, unfluidized
- No pH correction. Final pH around 7.2

The results, shown in Table 16, are promising. The removal efficiency is defined as the percentage of the ortho-phosphate that is removed from the water. For this test, this was >99%. Crystallization ratio is the percentage of the removed P that stays in the crystallizer and thus crystallizes on- or is otherwise captured by the bed. This part is recoverable as vivianite. For this test a crystallization ratio of 78% was achieved. The sand bed turned blue after recovery and exposure to air, indicating vivianite presence. (Figure 17)

Table 16. Crystallization results of test with new setup

Time	**O_2**	**pH**	**oP**	**Total P**	**Turbidity**	**Removal**	**Crystallization ratio**
	mg L^{-1}		mg L^{-1}	mg L^{-1}	NTU	%	%
15:10	1.32	8.4	19.4	19.5	23.8	0%	0%
15:25	0.38	7.2	<0.05	10.5	147	>99%	46%
15:40	0.36	7.23	<0.05	8.59	134	>99%	56%
16:10	0.34	7.29	<0.05	7.66	117	>99%	61%
16:40	0.34	7,.	<0.05	4.24	50.2	>99%	78%
17:10	0.34	7.18	<0.05	5.7	66.3	>99%	71%

Figure 17. Blue vivianite on sand after experiment with bench-scale setup

Further testing is currently being performed to reproduce these results for a lower influent P concentration (in the order of 5 mg L^{-1}, which will be the order of influent concentration for CS5). However, based on the findings so far, some design parameters have start to be defined, as presented in the next chapter.

3.2. Definition of the design parameters of the pilot units for CE processes

3.2.1. Granular AnMBR reactor

a) Influent characteristics

The granular AnMBR will be fed from the effluent of the primary settler of La Llagosta WWTP. Table 17 summarizes the physicochemical composition of the main components of this stream, which is in the range of the reported values for this kind of wastewaters (Metcalf and Eddy, 2003).

Table 17. Physicochemical composition of wastewater from La Llagosta WWTP to be treated by the granular AnMBR

Parameter	Value	Unit
Total chemical oxygen demand (COD)	600*	mg L^{-1}
Soluble chemical oxygen demand	180*	mg L^{-1}

Total suspended solids (TSS)	19**	mg L^{-1}
Volatile suspended solids (VSS)	19**	mg L^{-1}
Ammonium (N-NH_4^+)	47**	mg L^{-1}
Nitrite (N-NO_2^-)	Not detected	mg L^{-1}
Nitrate (N-NO_3^-)	Not detected	mg L^{-1}
Total nitrogen (TN)	Not determined	mg L^{-1}
Phosphate (PO_4^{3-})	3.9**	mg L^{-1}
Total phosphorous (TP)	5.1**	mg L^{-1}
Sulfate	364**	mg L^{-1}
pH	7.8**	-
Conductivity	1.1**	mS cm^{-1}
Turbidity	4**	NTU
Alkalinity	538**	mg$CaCO_3$ L^{-1}

Where: * estimated from hystorical data from La Llagosta WWTP and ** experimental data from subtask 5.1.1

b) Design calculations and considerations

The granular AnMBR consists in different process units, as schown in Figure 18. The granular AnMBR was designed to have a treatment capacity up to 10 m^3 d^{-1}. Considering the performance of the bench-scale test, the composition of the wastewater to be treated and flow-rate of 10 m^3 d^{-1}, a granular AnMBR was designed as follows.

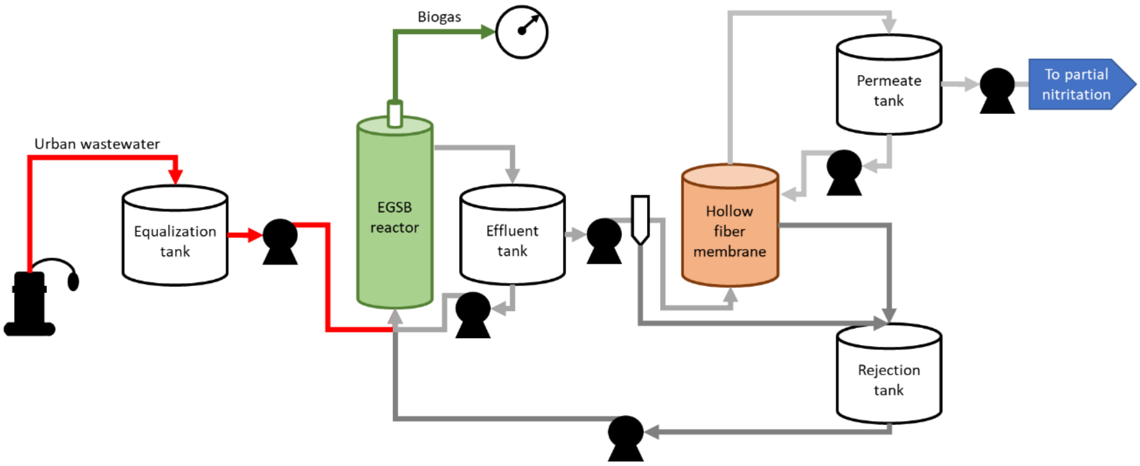

Figure 18. Scheme of the granular AnMBR at pilot scale.

The granular AnMBR consists in several units connected in series. Firstly, a submersible pump allows to fill an equalization tank. This deposit was selected to accumulate and ensure stability of the inflow-rate. This tank will vahe a volume of 2 m^3, connected to a centrifugal pump and pipelines to feed the wastewater to the anaerobic reactor, EGSB technology. A sensor level will switch ON/OF the submersible pump.

An EGSB reactor was selected as the most suitable technology to perform the anaerobic digestion with granular biomass at mainstream conditions (see Deliverable D5.1 and section 4.1.1 for more information). The reaction volume (V_R) of the EGSB was defined according to equation 1 and considering the OLR obtained in the bench-scale tests and the flow rates to be applied.

$$V_R = \frac{COD_0 \cdot Q_0 + COD_{RWW} \cdot Q_{RWW}}{OLR} \qquad \textit{Equation 1}$$

Where COD_0 is the COD concentration in the raw wastewater (kg m^{-3}), Q_0 is the inflow rate in the raw wastewater (m^3 d^{-1}), COD_{RWW} is the COD concentration in the reject wastewater from the hollow fiber membranes (kg m^{-3}), Q_{RWW} is the inflow rate in the reject wastewater from the hollow fiber membranes (m^3 d^{-1}) and OLR is the organic loading rate (in kgCOD m^{-3} d^{-1}).

The EGSB was designed as cylindrical shape, due to (i) space availability, (ii) easily to transport, (iii) better handly during installation, (iv) strongest geometrical structure than square/rectangular ones and (iv) economical aspects due to smaller surface area than rectangular surface area.

Knowing the shape and reaction volume of the reactor and considering the applied upflow velocity to be applied, the transversal area (A_R) and the diameter (D_R) of the reactor can be defined according to equation 2 and 3, respectively:

$$A_R = \frac{Q_{In}}{v_s \cdot 24} \qquad \textit{Equation 2}$$

Where Q_{in} is the total inflow rate fed to the reactor (m^3 d^{-1}), v_s is the upflow velocity applied (m h^{-1}) and 24 if the conversion factor from hours to days.

$$D_R = \sqrt{\frac{4 \cdot A}{\pi}} \qquad \textit{Equation 3}$$

Where A is the transversal area (m^2).

The height of the reaction colum (H_R) can be calculated accordind to Equation 4

$$H_R = \frac{V_R}{A} \qquad \textit{Equation 4}$$

The v_s applied was considered based on the data from the bench scale tests and avoiding an excessive hydraulic load, consequently, guarantee high biomass washout. Conventionally, EGSB reactor applies high upflow velocicites (> 5 m h^{-1}), however due to the (i) complexity of the urban wastewaters (high % of particulate matter), (ii) low temperatures in winter sessions and (iii) high height to diameter ratio, hydraulic limitantions can be considered.

After the reaction volume has been defined, the design of the EGSB reactor requires some technical consideration related to its internal parts: (i) the gas-liquid-solid (GLS) separator and (ii) inlet distribution. To design these parts, some considerations are required.

The GLS separator is one of the most critical parts of the design of an EGSB reactor, as it must provide (i) efficient gas separation from the liquid and solids phases, where it is entrapped, (ii) guarantee the settling of the granular biomass with entrapped gas bubbles and (iii) provide high quality of the treated wastewater by reducing the solid washout. To achieve these goals, enough surface area should be provided in the GLS separator. Its design depends on the caracteristics of the treated wastewater, organic loading rate, particle size and settling capacity of the biomass, biogas production and size and shape of the reactor. Due to the EGSB reactor has a cylindrical shape and small size, a conical shape of the SLG separator. The area for GLS separator (A_{GLS}) can be calculated according to Equation 5.

$$A_{GLS} = \frac{Q_{In}}{v_{s-max} \cdot 24} \qquad \textit{Equation 5}$$

Where v_{s-max} is the maximum upflow velocity that could reach to the GLS separator, taking into consideration the biogas production (m h^{-1}). The diameter of the GLS separator (D_{GLS}) can be calculated with equation 3. The applied V_{s-max} was 6.0 m h^{-1}.

The effluent of the EGSB reactor will be obtained by overflow through the top of the reactor, in this way, no extra pumping is needed, thus reducing the operational costs. The top of the reactor will be close, allowing the exit of the biogas through the GLS separator.

The biogas production will be quatified by a on-line volumetric gasmeter, located at the top of the reactor and connect to the gas effluent pipeline from the GLS separator.

Previous to the GLS separator, a reduced aperture was included to enhace the solid-liquid-gas separation and avoid malfunction of the settling compartment. For the design of the EGSB reactor a 65 % was considered, thus the diameter of the apperture was 0.33 m.

Inlet distribution is another key parameter in the design of an EGSB reactor. This fact releases on (i) to allow optimum contact between substrate and biomass and (ii) to avoid preferential flows and/or dead zone trough the sludge bed. The influent is introduced from the bottom of the reactor and distributed through 9 inlet distribution points, each of 10 mm diameter. In this way, homogeneous distribution of the wastewater is expected. In order to facilitate the maintenance of the reactor, the top and the bottom of the reactor are demountable.

Table 18 shows the design parameters of the EGSB reactor. Figure 19. Technical drawing of the EGSB reactor   shows the technical drawing of the EGSB reactor.

The EGSB reactor reactor will be construted in glass fibre reinforced polymer (GFRP). Beside it will be heat-insulated to avoid high fluctations of the reaction temperature.

Table 18. Design parameters of the EGSB reactor

Parameter	Value	Unit
Flow-rate of raw wastewater	10	$m^3\ d^{-1}$
Flow rate of the rejection from hollow fiber membrane	8.4	$m^3\ d^{-1}$
Total incomming flow-rate	18.4	$m^3\ d^{-1}$
OLR	10.7	Kg COD $m^{-3}\ d^{-1}$
Liquid upflow velocity	3.9	$m\ h^{-1}$
Reaction volume	0.8	m^3
Total volume	0.9	m^3
Shape of the reactor	Cylindrical	-
Diameter of the reactor	0.50	m
Diameter of the apperture prior to the GLS separator	0.33	m
Height of the reaction columm	4.25	m
Height of the effluent discharge	4.68	m
Total height of the reactor	4.78	m

Shape of the GLS separator	Cylindrical	-
Number of separators	1	-
Diameter of the GLS separator	0.40	m
Height of the GLS separator	0.35	m
Angle of the GLS separator	60	°
Number of inlet distributors	9	-
Diameter of the inlet distributor	10	mm
Number of sampling point	6	-
Distance between sampling points	1.00	m
Number of effluent withdrawal	1	-

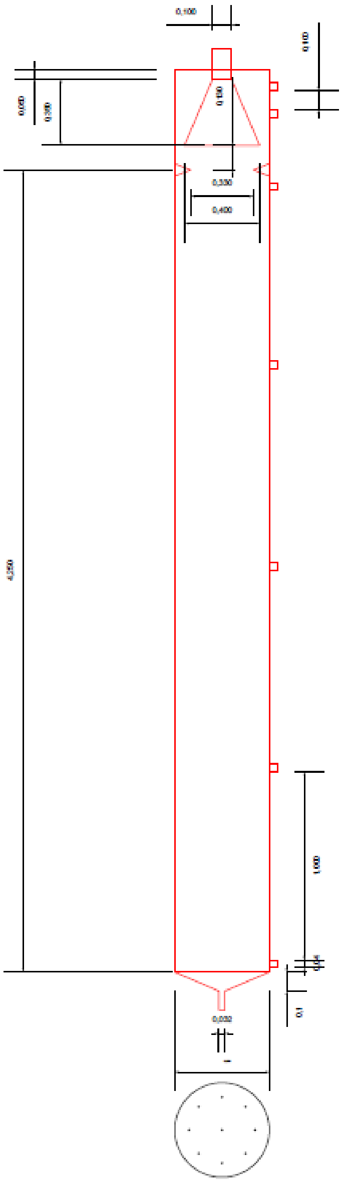

Figure 19. Technical drawing of the EGSB reactor

The temperature of the reactor will be monitored by an on-line sensor at the effluent pipeline. The effluent of the EGSB reactor will be collected and accumulated in a close tank of 1 m^3. For the start-up period, an centrifugal pump was included to feed to the reactor with the produced effluent (treated wastewater), in case of extra flow-rate is needed to achieve the design upflow velocity.

The produced and accumulated effluent of the EGSB reactor will be pumped to a ultrafiltration (UF) membrane, using hollow fiber technology from DuPont's company. Two modules of IntegraFlux SFP-2860XP will be installed, allowing to work with 102 m^2 of membrane area. Prior to the UF membrane, strainer/prefilter (with pore size higher than 200 µ) will be used to ensure safy conditions for the hollow fiber membranes and avoid excessive clogging due to particles higher than 200 µm. The prefilter washing will be automatic and reject stream will be accummulated in the rejection tank.

The hollow fiber membranes will be contained inside a PVC-case and will be operated in "dead end" configuration. This means that the effluent of the EGSB reactor will be fed continuosly the two filtration units, until the maximum operational pressure is achieved (1 bar). At these conditions, the permeate production is low and fouling of the fibers is achieved. Filtration will be done working as "out-in" mode, meaning that the raw wastewater pass through the fibers from the out side of the fiber to the inside of it. The produced permeate (stream with low concentration of solids) will be accumulated in the permeate tanks (2 deposits of 2 m^3). To clean the hollow fibers, a backwash period will be implemented at 100 LMH (i.e. 245 m^3 d^{-1}), feeding the produced permeate through the fibers. The backwash parameters has been suggested by the provider (see Table 19) and they will be checked/modified along the operation of the membranes. It was estimated that 8.4 m^3 d^{-1} of reject wastewater from the UF membranes (stream with high content of solids). Estimation was done using Water Application Value Engine (WAVE) software from DuPont. Reject stream will be accumulated in a rejection tank (1 m^3) and recirculated (at 8.4 m^3 d^{-1}) to the EGSB reactor. Thus, the total feeding flow rate to the EGSB will be 18.4 m^3 d^{-1}. The provider suggests to perform Chemically Enhanced Backwash (CEB) to boost the effectiveness of a backwash and remove impurities that can not be removed by only applying backwash. UF provider suggested to apply an acid CEB once per day and an alkali CEB every 6 h of operation (4 per day in total). Acid CEB will use HCl 32 % as reagent and alkali CEB will apply NaOH 50 % and NaOCl 12 %. CEB parameters are summarized in Table 20. Dosing pump will add the chemical reagents during CEB. The produced stream from the CEB will not be returned to the EGSB reactor, in order to avoid destabilization of the operation due to addition of chemical reagents. Under the applied conditions (200 LMH of backwash), 2 UF units and estimating a operational flux of 12 LMH, its expected to produce 10 $m^3\ d^{-1}$ of permeate to feed the partial nitritration reactor.

Table 19. Backwash parameters for the hollow fiber membranes (suggested by the provider)

	Forward flush	Filtration mode	Air inlet	Drain	Backwash 1	Backwash 2	Forward flush	Filtratiom mode
Duration	2.0-3.0 min	25.0 min	20 sec	30 s	30 s	30 s	85 s	25.0 min
Flow-rate	1.6 $m^3\ h^{-1}$	37 LMH	12.0 N $m^3\ h^{-1}$	By gravity	100 LMH	100 LMH	1.6 $m^3\ h^{-1}$	37 LMH

Table 20. Chemically Enhanced Backwash (CEB) parameters for the hollow fiber membranes (suggested by the provider)

	Duration	Flow-rate
Filtration mode	25 min	37 LMH
Air inlet	20 s	12.0 N $m^3\ h^{-1}$
Drain	30 s	By gravity
Backwash 1	30 s	100 LMH
Backwash 2	30 s	100 LMH
Soak	10.0 min	0.0 $m^3\ h^{-1}$
Air inlet	20 s	12.0 N $m^3\ h^{-1}$
Drain	30 s	By gravity
Backwash 1	30 s	100 LMH
Backwash 2	30 s	100 LMH
Forward Flush	85 s	1.6 $m^3\ h^{-1}$
Filtration mode	25.0 min	37 LMH

The raw wastewater, effluent from the EGSB reactor, rejection wastewater and backwash streams will be pumped by centrifugal pumps connected to variable-frequency drives and flowmeters for better control and monitoring of theses streams.

All the piping line connections between pumps, reactors and devices will be done with DN32 of unplasticised polyvinyl chloride (PVC-U). The design of the pilot plant considered that all the applied tanks will have an overflow security exit and connections for inlet, pumps and drain valve.

A Programmable Logic Controller (PLC) was included to achieve the diserable control loops for pumping and ultrafiltration sequences. Temperature will be monitored on-line. Pressure sensor on the inlet and outlet of the UF will allow automatic control of this process. The PLC will regulate the pumps.

3.2.2. Partial nitritation reactor

a) Influent characteristics

The effluent produced by the granular AnMBR will be treated by an partial nitritation reactor. The estimated composition of this stream is shown in Table 21, based on the bench-scale tests (see Deliverable D5.1 and section 4.1.1 for more information).

Table 21. Physicochemical composition of the effluent produced by the granular AnMBR to be treated by the partial nitritation reactor

Parameter	Value	Unit
Total chemical oxygen demand (COD)	296**	mg L^{-1}
Soluble chemical oxygen demand	54**	mg L^{-1}
Total suspended solids (TSS)	4***	mg L^{-1}
Volatile suspended solids (VSS)	4***	mg L^{-1}
Ammonium (NH_4^+)	50*	mg L^{-1}
Nitrite (NO_2^-)	6***	mg L^{-1}
Nitrate (NO_3^-)	6***	mg L^{-1}
Total nitrogen (TN)	70**	mg L^{-1}
Phosphate (PO_4^{3-})	5***	mg L^{-1}
Total phosphorous (TP)	6***	mg L^{-1}
Sulfate	154***	mg L^{-1}
pH	8***	-
Conductivity	2***	mg L^{-1}
Turbidity	4***	NTU

Where: * expected concentration, ** estimated from the operation of the bench-scale tests and *** experimental data from subtask 5.1.1.

b) Design calculations and considerations

The partial nitritation system consists in different process units as shown in Figure 20. This system was designed to have a treatment capacity up to 10 m^3 d^{-1}, considering the performance of the bench-scale tests, the composition of the wastewater to be treated and flow-rate of 10 m^3 d^{-1}. The partial nitritation (PN) reactor was designed as follows.

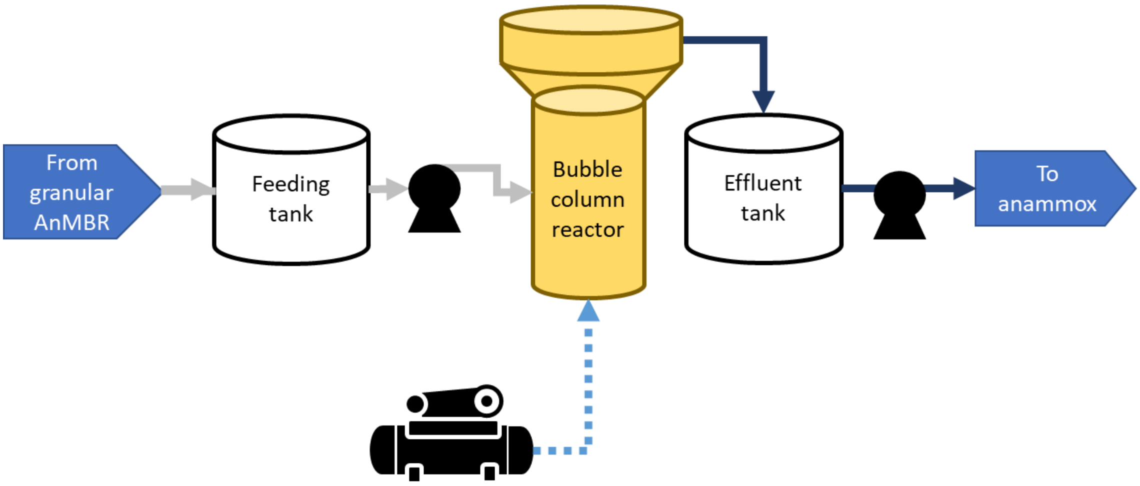

Figure 20. Scheme of the partial nitritation system at pilot scale.

The feeding tank allows to accumulate up to 2 m^3 of the produced effluent from the granular AnMBR, i.e. the permeate. A centrifugal pump feeds the bubble column reactor with the wastewater to be treated.

A bubble columm reactor will perform the partial nitritration process. Airflow will be introduced from the bottom of the reactor through 3 diffusers, to produce homogeneous distribution of the air and supply the required oxygen for biomass growth and mixing purposes. A GLS separator at the upper part of the reactor will allow an efficient separation of the granular biomass, the gases and treated wastewater. The bubble column reactor is divided in two parts, the body of the reactor and the in-built secondary clarifier at the top (head).

The reaction volume (V_R) of the bubble column reactor was defined according to equation 6 and considering the NLR obtained in the bench-scale tests and the flow rates to be applied.

$$V_R = \frac{N_0 \cdot Q_0}{NLR} \qquad \textit{Equation 6}$$

Where N_0 is the nitrogen concentration in the wastewater (kg m^{-3}), Q_0 is the inflow rate in the raw wastewater (m^3 d^{-1}) and NLR is the nitrogen loading rate (in kgN m^{-3} d^{-1}).

For the design, it was considered that the body of the reactor was 50 % of the reaction volume. Thus, the volumen of the GLS separator is 0.5 m^3 and the body 0.5 m^3.

As the EGSB reactor, the PN reactor was designed as cylindrical shape, due to (i) space availability, (ii) easily to transport, (iii) better handly during installation , (iv) strongest geometrical structure than square/rectangular ones and (v) economical aspects due to smaller surface area than rectangular surface area.

Knowing the shape and volume of the body and considering a defined height, the transversal area of the body (A_B) and the diameter of the body (D_B) can be defined according to equation 7 and 3, respectively:

$$A_B = \frac{V_B}{H_B}$$ *Equation 7*

Where V_B is the volume of the body (m^3) and HB is the height of the body (m).

The height of the reaction colum (H_R) can be calculated accordind to equation 4.

$$H_R = \frac{V_R}{A}$$ *Equation 4*

Considering that the height of the GLS separator is 25 % of the total height and this last value is 2 m, the height of the GLS separator can be estimated, which was 0.5 m.

The design of the GLS separator is critical to the performance of the reactor, as it must provide (i) efficient gas separation from the liquid and solids phases, (ii) guarantee the settling of the granular biomass and (iii) provide high quality of the treated wastewater by reducing the solid washout. To achieve these goals, enought surface area should be provided in the GLS separator. Its design depends on the caracteristics of the treated wastewater, nitrogen loading rate, particle size and settling capacity of the biomass, aeration rate and size and shape of the reactor. Due to the PN reactor has a cylindrical shape and small size, a conical shape of the SLG separator. To supply enough area, the diameter of the GLS separator was 70 % higher than the diameter of the body, thus it was designed for 1.20 m. The inner parts of the GSL separator considered the diameter of the body.

The effluent of the PN reactor will be obtained by overflow through the top of the reactor, in this way, no extra pumping is needed, thus reducing the operational costs. The top of the reactor will be open.

The incoming air will be introduced by the bottom of the reactor and it will be controlled, by a rotameter and a variable-frequency drives connected to an air compressor. Air will enter through 3 air difussers of 9 inches.

Table 22 summarizes the design parameters of the EGSB reactor. Figure 21 shows the technical drawing of the bubble column reactor.

The bubble column reactor will be construted in GFRP. Besides, the reactor will have a temperature control system to maintain 30 ºC (optimum temperature for NP process) during start-up period, by means of heat-exchanger connected to the reactor. Also, it will be heat-insulated to avoid high fluctations of the reaction temperature.

The design of the bubble column reactor included several automatic loops regulated by a PLC. Temperature, pH, dissolved oxygen and ammonium/nitrate probes will be implemented. These parameters will be monitored and controlled for better performance of the partial nitritation process. Moreover, the PLC will allow the operation of the PN reactor as sequencing batch reactor (SBR) during the start-up to form granular biomass from flocs (inoculum). To achieve this SBR operation, the PLC will control the pump, drain valve and aeration loops.

The effluent of the bubble column reactor will be collected and accumulated in a close tank of 1 m^3. The produced effluent of the PN reactor will be pumped to a next process: anammox.

The wastewater to be treated and produced effluent streams will be pumped by centrifugal pumps connected to variable-frequency drives and flowmeters for better control and monitoring of theses streams. All the piping line connections between pumps, reactors and devices will be done with DN32 of PVC-U. The design of the pilot plant considered that all the applied tanks will have an overflow security exit and connections for inlet, pumps and drain valve.

Table 22. Design parameters of the bubble column reactor

Parameter	Value	Unit
Flow-rate	10	$m^3\ d^{-1}$

Nitrogen loading rate	0.5	Kg N m^{-3} d^{-1}
Air flow-rate	70-250	L min^{-1}
Reaction volume	1.0	m^3
Total volume	1.2	m^3
Shape of the reactor	Cylindrical	-
Diameter of the reactor	0.70	m
Diameter of the apperture prior to the GLS separator	0.70	m
Height of the reaction columm	1.28	m
Height of the effluent discharge	1.78	m
Total height of the reactor	2.00	m
Shape of the GLS separator	Cylindrical	-
Number of separators	1	-
Diameter of the GLS separator	1.20	m
Diameter of the out-side GLS separator	0.70	m
Diameter of the in-side GLS separator	0.35	m
Height of the GLS separator	0.25	m
Angle of the GLS separator	60	°
Number of inlet distributors	9	-
Diameter of the inlet distributor	10	mm
Number of sampling point	6	-
Distance between sampling points	0.40	m
Number of effluent withdrawal	1	-
Number of air diffusers	5	-
Diameter of the air diffuser	12	inchs

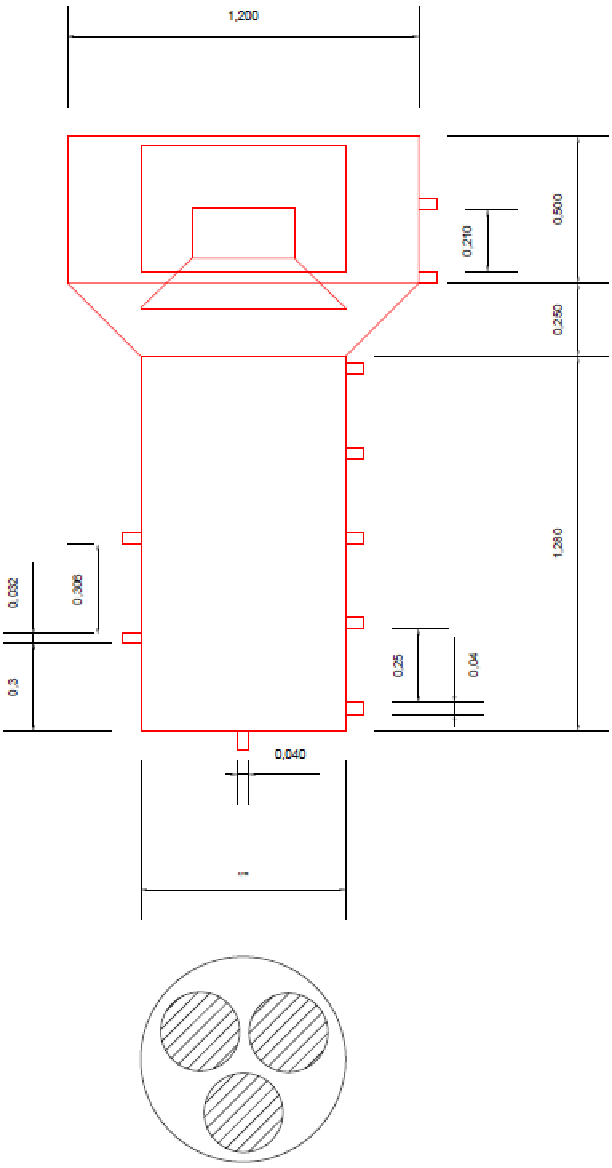

Figure 21. Technical drawing of the bubble column reactor.

3.2.3. Anammox reactor

a) Influent characteristics

After the partial nitritation process, an autotrophic denitrification via anammox biomass will be implemented to treat the produced effluent and achieve the nitrogen removal. The estimated composition of this stream is shown in Table 23, based on the bench-scale tests (see Deliverable D5.1 and section 4.1.1 for more information).

Table 23. Physicochemical composition of the effluent produced by partial nitritration reactor to be treated by the anammox reactor

Parameter	Value	Unit
Total chemical oxygen demand (COD)	Not determined	mg L^{-1}
Soluble chemical oxygen demand	Not determined	mg L^{-1}
Total suspended solids (TSS)	Not determined	mg L^{-1}
Volatile suspended solids (VSS)	314 **	mg L^{-1}
Ammonium (NH_4^+)	20*	mg L^{-1}
Nitrite (NO_2^-)	25*	mg L^{-1}
Nitrate (NO_3^-)	5**	mg L^{-1}
Total nitrogen (TN)	Not determined	mg L^{-1}
Phosphate (PO_4^{3-})	Not determined	mg L^{-1}
Total phosphorous (TP)	5.0*	mg L^{-1}
pH	7.6***	-
Conductivity	Not determined	mg L^{-1}
Turbidity	Not determined	NTU

Where: * expected concentration, ** estimated from the operation of the bench-scale tests and *** experimental data from subtask 5.1.1.

b) Design calculations and considerations

The selected technology was an Upflow Anaerobic Sludge Blanket (UASB) reactor. The UASB reactor will be fed with the effluent from the PN system. As the EGSB reactor, this technology includes a GSL separator on the top of the reactor and a tank where the wastewater flows in an upward direction through an anaerobic sludge bed.

The anammox process consists in different process units as shown in Figure 22. The Anammox UASB was designed to have a treatment capacity up to 10 m^3 d^{-1}. Considering the performance of the bench-scale test, the composition of the wastewater to be treated and flow-rate of 10 m^3 d^{-1}, the UASB reactor was designed as follows.

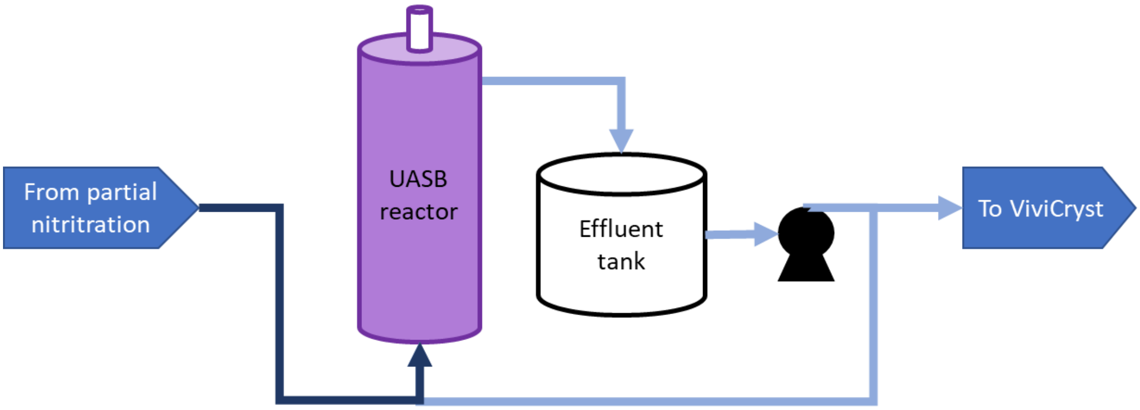

Figure 22. Scheme of the anammox system at pilot scale.

The reaction volume (V_R) of the UASB reactor was defined according to equation 6. As the UASB reactor is similar in several aspected to the EGSB reactor, the same most of the applied equation can be implemented for the design of this reactor. The UASB reactor was designed as cylindrical shape, as previously indicated.

Knowing the shape and reaction volume of the reactor and considering the applied upflow velocity to be applied, the transversal area (A_R) and the diameter (D_R) of the reactor can be defined according to equation 2 and 3, respectively. With this information, the height of the reaction colum (H_R) can be calculated accordind to Equation 4.

After the reaction volume was been define, as the EGSB reactor, the design of the UASB reactor requires some technical consideration related to its internal parts: (i) the gas-liquid-solid (GLS) separator and (ii) inlet distribution. The same considerations for thEGSB can be considered for the UASB reactor. Due to the EGSB reactor has a cylindrical shape and small size, a conical shape of the SLG separator. The area for GLS separator (A_{GLS}) can be calculated according to Equation 5. The applied $V_{s\text{-}max}$ was 3.0 m h^{-1}. The effluent of the UASB reactor will be obtained by overflow through the top of the reactor, in this way, no extra pumping is needed, thus reducing the operational costs. The top of the reactor will be close, allowing the exit of the produced nitrogen gas through the GLS separator.

Previous to the GLS separator, a reduced aperture was included to enhace the solid-liquid-gas separation and avoid malfunction of the settling compartment. For the design of the EGSB reactor a 36 % was considered, thus the diameter of the apperture was 0.25 m.

Inlet distribution is another key parameter in the design of an UASB reactor. This fact releases on (i) to allow optimum contact between substrate and biomass and (ii) to avoid preferential flows and/or dead zone trough the sludge bed. The influent is introduced from the bottom of the reactor and distributed through 9 inlet distribution points, each of 10 mm diameter. In this way, homogeneous distribution of the wastewater is expected. In order to facilitate the maintenance of the reactor, the top and the bottom of the reactor are demountable.

The design parameters of the UASB reactor is summarized on Table 24, and Figure 23 shows the technical drawing of the UASB reactor.

This reactor will be constructed in glass fibre reinforced polymer. Besides, the reactor will be heated to maintain optimum temperature of the process (35ºC), during the start-up period. To achieve this, a heat-exchanger connected to an effluent recirculation pump will be implemented. Moreover, the reactor will be heat-insulated to avoid high fluctations of the reaction temperature. The PLC will control the automatic loop for temperature control and the pumps for feeding and recirculation of the effluent.

The effluent of the UASB reactor will be collected and accumulated in a close tank of 1 m^3. For the start-up period, an centrifugal pump was included to feed to the reactor with the produced effluent (treated wastewater), in case of extra flow-rate is needed to achieve the design upflow velocity. The produced effluent of the UASB reactor will be pumped to the next process: ViviCryst.

The wastewater to be treated and produced effluent streams will be pumped by centrifugal pumps connected to variable-frequency drives and flowmeters for better control and monitoring of these streams. All the pipine line connections between pumps, reactors and devices will be done with DN32 of PVC-U. The design of the pilot plant considered that all the applied tanks will have an overflow security exit and connections for inlet, pumps and drain valve.

Table 24. Design parameters of the UASB reactor

Parameter	Value	Unit
Flow-rate	10	$m^3\ d^{-1}$
NLR	0.5	$kgN\ m^{-3}\ d^{-1}$
Liquid upflow velocity	1.0	$m\ h^{-1}$
Reaction volume	1.1	m^3
Total volume	1.2	m^3
Shape of the reactor	Cylindrical	-
Diameter of the reactor	0.70	m
Diameter of the apperture prior to the GLS separator	0.25	m
Height of the reaction columm	2.60	m

Height of the effluent discharge	3.24	m
Total height of the reactor	3.34	m
Shape of the GLS separator	Cylindrical	-
Number of separators	1	-
Diameter of the GLS separator	0.58	m
Height of the GLS separator	0.50	m
Angle of the GLS separator	60	°
Number of inlet distributors	9	-
Diameter of the inlet distributor	10	mm
Number of sampling point	6	-
Distance between sampling points	0.65	m
Number of effluent withdrawal	1	-

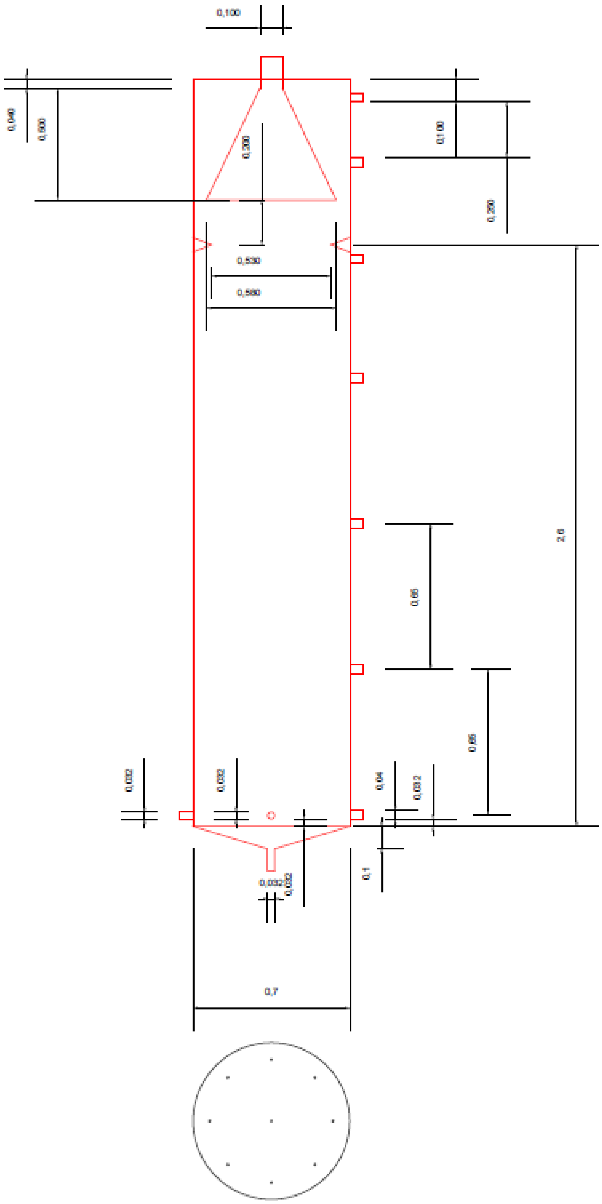

Figure 23. Technical drawing of the UASB reactor

Figure 24 shows the piping and instrumentation diagram (PI&D) of the pilot plant including the granular AnMBR, partial nitritation and anammox processes. The anaerobic process will be installed in a marine contained of 40 feet and the partial nitritation followed the anammox will be inside a marine container of 20 feet.

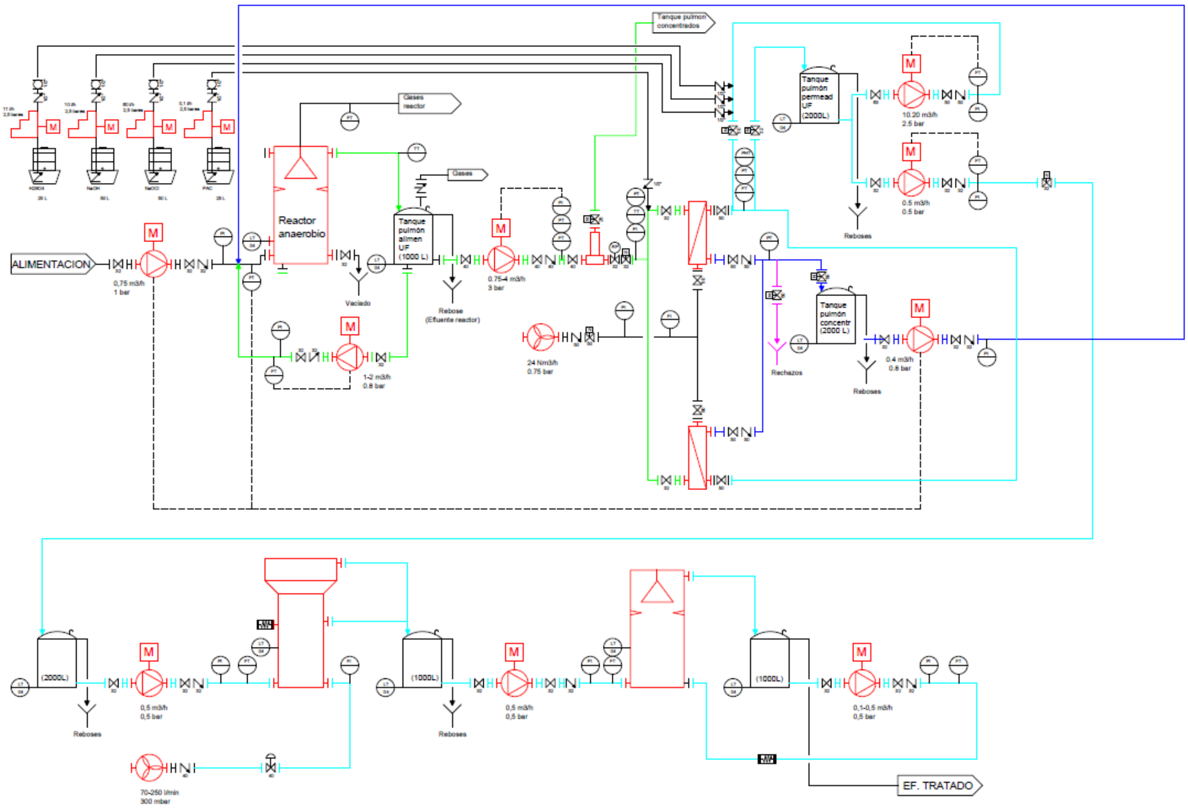

Figure 24. PI&D of the pilot plant to be constructed to implement the granular AnMBR, partial nitritation and anammox processes.

3.2.4. ViviCryst technology

Figure 25 shows a scheme of the ViviCryst installation at pilot scale.The design parameters of the ViviCryst pilot installation have not been finalized yet at the time of writing and are subject to change. However, based on our findings with bench-scale experiments so far, some parameters can start to be defined.

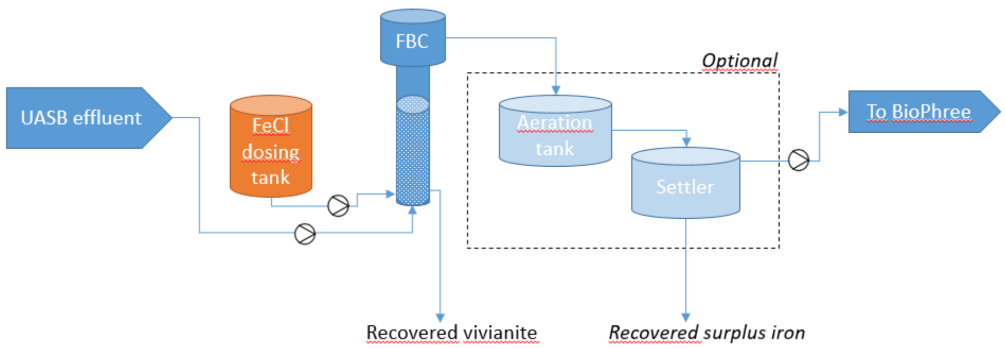

Figure 25. Scheme of the ViviCryst pilot installation

For instance, the upflow velocity to have a fluidized bed will be in the range of 30 to 40 m h^{-1}. To achieve this upflow with only the influent flow, a reactor diameter of 11-13 cm should be used.

Using this diameter range will result in a loading of 0.15 – 0.2 kgP m^{-2} h^{-1} with an influent concentration of 5 mg L^{-1} P. From the performed tests, it can be seen that a low loading is beneficial for crystallization ratio. However, further tests need to be done to confirm that this value will work.

Another relevant design parameter is hydraulic retention time (HRT) in the reactor. Bench-scale tests used a HRT of 6 minutes, but it is likely that this parameter can be decreased. At bench-scale tests over 99% of the P was removed, but it would be preferable to achieve a lower P removal and obtain an effluent with 1 mg L^{-1} P to be removed in a later stage by BioPhree technology. The latter, because crystallization rate is 78%, therefore the rest of removed P gets lost as very fine particulate P that cannot be recovered afterwards with BioPhree.

Assuming an HRT of 6 minutes, the reactor height will be 3 meters. If a lower HRT of 4 minutes is chosen, the reactor height will be 2 meters.

Other important design parameters and considerations:

- ViviCryst will be a containerized pilot for easy transport
- Basic equipment will consist of the reactor (11-13 cm diameter and 2-3 meter high), an upflow pump (400 L h^{-1}), an iron dosing pump, a settler unit for capturing fines in the effluent or/and an effluent buffer tank. It will also include sensors, sampling points, PLC, among others.
- Optional features: pH correction in the influent, and aeration unit for the effluent (this would allow iron recovery turning Fe^{2+} into Fe^{3+}, which has lower solubility and would precipitate as FeOH)
- Seed bed: Needs a way to be harvested and replaced periodically
- Upflow velocity: To be controlled and kept constant with recirculation of (filtered) effluent
- Iron dosing: At the bottom of the seed bed in form of concentrated FeCL solution

Concluding remarks

The design as well as the construction of the pilot units for CS4 described in this deliverable has been successfully completed. Bench-scale results and previous knowledge about the processes were the basis for developing the design of these units. The pilot units were transported to Larnaca (Cyrpus) in December 2021, and the start-up was completed during January 2022. During the following months, the pilot units will be operated in an integrated manner, to optimize the recovery of phosphorus, water and salts.

The design of the reactor pilot units was successfully completed and described in this deliverable. The AnMBR (EGSB selected based on the bench-scale results), the PN reactor and the Anammox reactor were designed considering the performance of the bench-scale test, the composition of the wastewater and a treating capacity of 10 $m^3\ d^{-1}$. Bench-scale tests performed previously were essential for the design process. The units are currently under construction, the expected delivery date is April 2022. Regarding the ViviCryst unit, the preliminary design was presented, but the final design is not completed yet. Recent design upgrades include the removal of the recirculation from the process. Wetsus will be focused on further optimizing the design during the following months, and the construction of the unit will start according to the planning (start-up of the unit is planned for October 2022).

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