

WALNUT

D2.8 - Pilot plant demo definition

WP2, T2.7 WalLAB methodology to scale-up technologies to pilot level

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Executive Summary

In this deliverable is included a summary of the results obtained in the tasks of nutrient recovery technology at lab scale, focusing on the best combinations of technologies and feedstocks. In these deliverable comparisons between different technologies with the same aim, different feedstocks, pre-treatments, adsorbents or regenerants are included. Furthermore, in this analysis is also included an LCSA perspective because it is not only important the technical part, also the environmental one must be taken into account. Merging and analysing all this information, different decisions and conclusion have been reached in order to scale-up these technologies and define the technologies, layout and operation aspects of the future WP3 pilot plants

Keywords: Brine, nutrient recovery, nitrogen recovery, adsorbent, microbial fuel cell, microalgae, ABC-BioPhosphate.



1. Introduction

1.1 Purpose, scope and target group

This deliverable, D2.8, as the last one of the Work Package 2 (WP), gathers the information of the different tasks involved in the WP mainly the technical conclusions of the different lab-scale technologies tested. These conclusions remark the key parameters for the technology scale-up which is going to take place in WP 3 which is the main aim of this deliverable. The target group of this deliverable are the partners involved in WP3 and WP5 due to these results are the first step for the development of these WP. Furthermore, out of the project, could be interesting for technological suppliers and industries who have the feedstocks used in these technologies as a stream in their processes.

1.2 Contribution partners

As is presented previously, this last deliverable collects the research carried out in tasks 2.2-2.6 related to different technologies for nutrient recovery at lab-scale. This task is led by CETAQUA and thus, CETAQUA guided the deliverable. The partners involved have been CETAQUA, CARTIF, Veolia, UGent, AQUAFIN, 3R, NTUA SDU and UC. CETAQUA, CARTIF, UGent, AQUAFIN, 3R and NTUA collaborate providing the technical support of their lab-scale technologies and writing their technical information. Furthermore, SDU and UC provided the preliminary information about the impact of the different technologies tested in this WP.

Table 1 shows an abstract of the contributions.

Table 1-1: Contribution of partners

Partner	Contribution
CETAQUA	Lead the task and the deliverable as well as technical contribution of Ion exchange and membrane contactors processes.
CARTIF	Technical contribution of Hybrid system of photoautotrophic and heterotrophic microalgal cultivation and Microbial Fuel Cell.
Veolia	Technical contribution of microbiological processes
UGent	Technical contribution of HRAS/CS + adsorption/ion-exchange
AQUAFIN	Technical contribution of HRAS/CS + adsorption/ion-exchange
3R	Technical contribution of ABC-BioPhosphate: multi functional biofertiliser and adsorbent
NTUA	Technical contribution of Nanofiltration and selective crystallisation processes
SDU	Preliminar LCSA perspective of the technologies
UC	Preliminar LCSA perspective of the technologies

1.3 Relation to other activities in the project

This deliverable gathers the information of the different lab-scale technologies in order to define the scale-up conditions as a first step to carry out the design, construction and operation of the different pilot plants. Thus, this deliverable is extremely related with Work Package 3 as well as Work Package 5 in which the environmental analysis is being carried out.

Table 1-2: Relation to other activities in the project

Task	Description
T3.2	Design, construction, installation and operation of the Pilot plant 1
T3.3	Design, construction, installation and operation of the Pilot plant 2



T3.4	Design, construction, installation and operation of the Pilot plant 3
T3.5	Design, construction, installation and operation of the Pilot plant 4
T3.6	Design, construction and installation of the Pilot plant 5
T5.2	Inventory analysis, modelling environmental emissions and soil fertility improvements and upscaling of technology performances



2. Deliverable overview

WalNUT project aim is to develop, assess and test 5 new integrated and sustainable technological solutions for highly efficient and effective NR from 5 waste water (WW) streams (urban, industrial, food, sewage sludge, brine from water desalination and demineralisation plants). Thus, by the cutting-edge sustainable innovations proposed in terms of techno-economic feasible solutions and safe and high-quality products (BBFs) from different WW streams, WalNUT will assure BBFs' public and regulatory acceptance and market incursion, promoting a circular economy context towards the replacement of non-renewable mineral fertilisers in the EU agricultural sector.

In this context, at this stage of the project, the main progress has been focused on the development of different technologies which have been tested at lab-scale as a first step towards their scale-up. Many different trials have been carried out working with different feedstocks, layouts and/or technologies looking for the best performance of each task. As a consequence of this research, this deliverable came up in order to gather the main conclusions obtained from tasks 2.2-2.6 from a technical point of view and incorporate environmental aspects to choose the best option and the best conditions to scale-up.

In the following sections, the different technologies and the options evaluated are going to be presented and analysed following the previous tasks carried out which are:

- Task 2.2 Microbiological processes (CARTIF and Veolia).
- Task 2.3 HRAS/CS + adsorption/ion-exchange (UGent and AQUAFIN).
- Task 2.4 ABC-BioPhosphate: multi-functional biofertiliser and adsorbent (3R).
- Task 2.5 Nanofiltration and selective crystallisation processes (NTUA).
- Task 2.6 Ion exchange and membrane contactors processes (CETAQUA).



3. Microbiological process

Microbiological processes are interesting alternatives for the recovery of nutrients from waste water. Thus, consortiums of microorganisms promote the consumption or fixation of organic matter to which they add the recovery of valuable inorganic elements such as N, P or K. Among all the possibilities, two microbiological processes seem more attractive due to the properties of the final product or the characteristics of each of them, as well as the purification capacity of the waste water used as raw material. These are nutrient recovery by growth of photosynthetic microorganisms (microalgae) and nutrient recovery by Microbial fuel cell (MFC).

Photosynthetic microorganism cultures can be applied as waste water treatment technology in photo-bioreactors. These systems offer several advantages over conventional water treatment processes, including the recovery of nutrients and CO₂ through autotrophic or heterotrophic metabolism. When consortia of photosynthetic microorganisms (usually green microalgae or cyanobacteria) and bacteria are employed, syntrophic processes concur to waste water treatment. On one side, bacteria oxidise organic carbon, enriching the medium of CO₂/HCO₃⁻ and nutrients, whereas photosynthetic microorganisms supply O₂ produced by photosynthesis to aerobic bacteria, while maximising inorganic nutrients uptake and recovery. Depending on the type of waste water, large amounts of K, N, and P are usually present, together with trace elements (B, Cu, Zn, Mo, Fe, Co and Mn), required for the growth of microalgae. In these systems, mechanical aeration to stimulate organic carbon oxidation could be avoided.

On the other hand, MFC is currently considered a promising technology in waste water treatment due to its production of electricity and waste water purification. Typically, the MFC has an anode chamber and a cathode chamber, respectively, and a cation exchange membrane (CEM) is installed to separate the two chambers. In the anode chamber, the anaerobic microorganisms are catalysts that convert chemical energy stored in organics directly into electricity. MFC can use sewage sludge as a substrate, which is considered a kind of biomass. Recently, nutrients recovery through the MFC process has attracted much attention since the energy recovered in the MFC may be used for its own operation and maintenance, which indicates that the MFC may be a neutral/positive energy balance system.

CARTIF and VEOLIA have tested both microbiological processes in WP2 at laboratory level (Hybrid system of photoautotrophic and heterotrophic microalgal cultivation and Microbial Fuel Cell). For each process, a design of experiments (DOE) was developed to determine the influence of the main operating parameters on the process. The experiments were carried out at CARTIF's laboratories using synthetic and real waste water from different industrial WWTP provided by VEOLIA. Finally, the results were analysed and compared to discern which one of the two technologies has a higher potential to be scaled up in WP3.

3.1 Alternatives

The final selection of the most promising technology between the two microbiological processes above tested was carried out considering the most decisive parameters for its scalability, its technical and economic profitability and its environmental sustainability. These parameters were: location and type of waste water used as feedstock, amount (in terms of percentage) of nutrient recovered, quality of the final effluent, operation time and energy consumed. In the following sections, the influence of each of the above parameters for the hybrid system of photoautotrophic and heterotrophic microalgal cultivation (microalgae technology) and Microbial Fuel Cell technology (MFC technology) will be addressed.

3.1.1 Feedstock

Regarding the type of waste water, between CARTIF and VEOLIA (partner in charge of the supply of waste water for the experiments and that in the future will operate the pilot 1 developed in WP3), they selected 3 types of waste waters out of a total of 5 in the initial screening, according to their characteristics). The characteristics of each of the waste water types considered are presented in Table 3-1.

Table 3-1.Characteristics of different waste water streams.



	Kind WW	Location (in Spain)	Flow (m ³ /d)	DQO (mg/L)	TS (mg/L)	Conductivity (mS/cm)	pH
WWTP 1	Bakery	South	42.75	11150.74	2747.83	5.26	8.83
WWTP 2	Sauces	East	120.21	8971.91	856.57	2.29	6.70
WWTP 3	Dairy	West	295.00	2224.05	420.00	2.81	11.05
WWTP 4	Brewery	South	1245.13	4619.07	1026.40	1.98	8.15
WWTP 5	Plastic waste recycling	West	40.95	2653.95	2301.98	2.34	7.26

The waste water streams presenting the highest organic matter content, the highest nutrient (N and P) concentration and the lowest concentration of Potentially Toxic Elements (PTEs) were selected.

From the five previous options the WWTPs number 1 and 2 were ruled out because of their lack of interest for NR practices, due to their low concentration of nitrogen (first one) and phosphorus (second one) in the influent of the plants. The other WWTPs' (WWTP 3, 4 and 5) influents were most suitable for NR purposes, so these plants' waste water was selected to be proved in WalLAB experiments.

The processes carried out in WWTPs 3, 4 and 5, respectively, are described below, Figure 3-1 also provides and overview of each WWTP and their location is included in Figure 3-2. The three processes are composed by a pre-treatment (WW conditioning stage), a primary treatment and a secondary biological treatment, but some unit operations are different:

- In WWTP 3, waste water is discharged by a dairy industry whose main activity is milk processing. There, water is basically used in equipment cleaning processes, and then it goes to the WWTP. In the WWTP, first WW is neutralised, as it usually arrives with pH values higher than 11, and coagulants and flocculants are added before a “Dissolved Air Flotation (DAF)” unit where, mainly, fats and oils are removed. Finally, the secondary treatment is an aerobic “Sequential Bioreactor_(SBR)” where the rest of COD and nutrients are removed from the WW stream.
- In WWTP 4, the stream treated is the effluent of a beverage industry, in this case, a brewery. The WW goes into the plant and, as in the other plant, is neutralised (if necessary) and coagulants and flocculants are added. After that, the stream passes through a primary settler where solids are separated, and then it goes to an anaerobic bioreactor to remove COD and nutrients. At the end, a secondary settler is installed to retain the microorganisms that might escape the bioreactor and return them to the bioreactor.

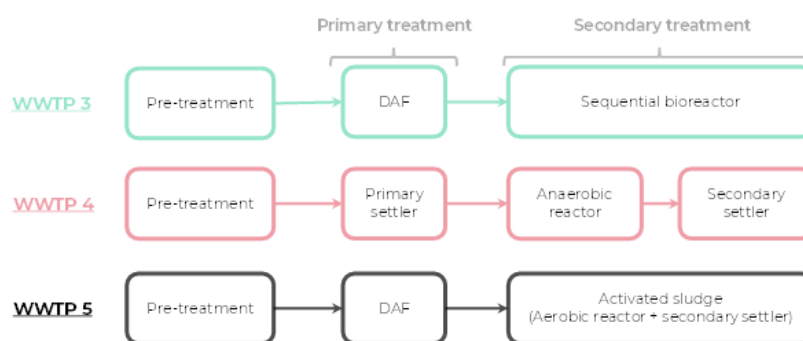


Figure 3-1. General description of the treatment system of the WWTPs.

- WWTP 5 is located in the premises of a recycled plastic production industry and treats an effluent with possible low amounts of plastic solids. Again, the WW can be neutralised (pH near to 7, usually) and



coagulation and flocculation reagents are added. A DAF is also installed to remove low density solids and a common activated sludge unit (aerobic reactor + secondary settler) follows it.

As mentioned before, in the WalLAB experiments, the WW came from these three plants in order to compare different feedstocks and select the most suitable for the microalgae process. It is important to highlight that the WW used was taken after the primary treatment of each plant. The purpose of that is to avoid extreme pH situations and non-soluble solids, which cannot be assimilated, in order to maintain the best possible conditions for microalgae cultivation.



Figure 3-2. Location of the selected WWTPs.

3.1.2 Nutrient recovery yield

One of the main aspects to be evaluated in the two technologies that will be crucial for the selection of the final technology is the nutrient recovery yield in each process. Thus, depending on the initial concentration of nutrients present in the waste water used as a raw material, the amount of N and P was determined. The amount of P and N in the final product was also evaluated. The yield is calculated in terms of nutrient in the final product divided by nutrient in the original waste water. This is a determining factor because one of the main objectives of the development of the technologies in the project is to maximise the production of fertiliser and its composition, so the higher the nutrient recovery yield, the greater the technical and economic viability of the process.

In the case of the two technologies developed by CARTIF in WP2 with the collaboration of VEOLIA, the nutrient recovery yield has been defined as one of the output variables of the DOE of the microalgae technology and the MFC technology. According to deliverable D2.3 in the microalgae technology, nutrient recovery yields of around 90 % (for TN) and 98% (for P) have been achieved, however, for the MFC technology; yields of 20% could not be exceeded.

3.1.3 Operation time

Reaction time influences technical development of the processes as well as the economics of the processes. In other words, the longer the residence time of the nutrient recovery process using microalgae, the greater the volume and number of bioreactors required, and the higher energy consumed for lighting the photobioreactor and aerating the heterotrophic bioreactors to carry out the cultivation in an optimal way and achieve high recovery yields and an adequate final effluent quality. With regard to the MFC process, the residence influences the volume of the microbial cells used to carry out the electrochemical process, as well as the amount of nutrient recovered, the degree of waste water purification (quality of the final effluent) and the energy generated, since as the residence time increases, each of the parameters indicated above increase. According



to the results obtained in the study of both technologies, the optimum residence time for the microalgae technology is around 3 days, while for the MFC technology it is 7 days.

3.1.4 Energy consumption

Energy consumption is often one of the determining factors for the successful scale-up of a process. Thus, the electrical energy demand of a process will influence the design of the process, as well as its economic profitability. In the processes under study, an intensive energy input was necessary in the microalgae technology. Firstly, artificial light was provided for the growth of the inoculum in the photobioreactor (by means of a led lamp between 6,000-9,000 lux) and the aeration. On the other hand, the heterotrophic stage of the algal culture was carried out by dark fermentation in closed bioreactors, so no light was needed, but energy was provided for agitation and aeration of the bioreactors.

Regarding the MFC technology, energy input was necessary for the agitation of the waste water inside the cell. This agitation was carried out by pumping and mechanical agitation (stirring plate), however, one of the great advantages of this technology is that it is a bioelectrochemical process, therefore, at the same time as the degradation of the organic matter of the waste water by the microorganisms was carried out, electrical energy was generated. Specifically, up to 5 mA and 500 mV of electrical energy was generated when about 600 mL of waste water was treated.

3.1.5 Final effluent quality

Although the main objective of the project is to develop safe bio-based fertilisers from waste water that can be marketed in the future, it is necessary that the nutrient recovery processes that generate the bio-based fertilisers in turn carry out waste water purification stages. Therefore, the design of the processes was carried out under the postulates of the circular economy and a final effluent was obtained with characteristics in accordance with legislation to be reused, apart from the main bioproducts.

In the case of the nutrient recovery process using microalgae, during the 3 days of operation, the COD of the waste water was reduced by 87%, with final COD values < 100 mgO₂/L (below the limit set by law). However, in the case of the MFCs process, the COD was only reduced by 80%, with final COD values of 800 mg O₂/L (above the legal limit for reuse).

3.2 Comparison

3.2.1 Feedstock

Taking into account the experiments carried out for the 3 types of waste waters tested at laboratory scale, the waste water with the best results for both types of technologies (microalgae and MFCs) has been the waste water from WWTP 3. This waste water is not only the best option for the results with respect to the microbiological processes but also due to logistical factors. Pilot 1 will be constructed at the CARTIF facilities and then shipped to WWTP 3 where it will be operated by VEOLIA, therefore, transport costs and all other issues concerning the installation will be simpler than in the other options considered.

3.2.2 Nutrient recovery yield

According to section 3.1.2, the nutrient recovery yield is much higher for the microalgae production technology; therefore, this was one of the most decisive factors for the selection of the technology. Having a high nutrient recovery rate will not only ensure a better nutritional composition of the biofertiliser obtained as a final product, but also a higher purification efficiency that will result in a higher quality of the final effluent reducing its environmental impacts like eutrophication potential.



3.2.3 Operation time

The operating time for both technologies has been fixed taking as a reference the time necessary to reduce the organic load of the waste water considerably and at the same time recover most of the nutrients from the water. The objective of this action was to check if the purification and nutrient recovery process allows on the one hand obtaining a significant amount of biofertiliser and on the other hand a composition of the final effluent so that it can be reused in accordance with legislation. For the microalgae technology it was possible to obtain in the effluent a COD below the legal limit for reuse and a high nutrient recovery rate (90-98 %) after 3 days of operation. However, for the MFCs technology, after 7 days of operation it was not possible to lower the organic load below the limit and a low nutrient recovery rate (20 %) was obtained. Therefore, the microalgae technology option shows better results with respect to the operation time.

3.2.4 Energy consumption

The major difference between the two technologies is that while the microalgae technology consumes thermal and electrical energy in the process, the MFC technology generates electrical energy. While scaling up the microalgae process would be done using renewable energies (solar thermal and biomass), making it self-sustainable, the MFC technology is more advantageous from an energy point of view.

3.2.5 Final effluent quality

As discussed in previous sections, microalgae technology achieved much better results in terms of final effluent quality than MFC technology. Microalgae treatment showed a high nutrient recovery rate and an organic load content below the limit in a shorter amount of time.

3.2.6 Environmental comparison

Besides the technical and economical aspect, it is important to evaluate the environmental aspects of each process. With the microalgae technology, there has been no consumption of reagents or consumables, while in the MFCs, in each experiment carried out, the consumption of electrodes (graphite, platinum, etc.) and Nafion™ for the exchange membrane between the cell chambers. However, in terms of energy consumption, as already mentioned in sections 3.1.4 and 3.2.4, the most favourable process was the MFCs. In terms of process costs, the assembly of the MFCs involved a higher cost (electrochemical cells, electrodes, membranes, etc.) than the two bioreactors necessary in the microalgae treatment process. Last but not least, the aspect of transport is paramount in the environmental assessment of the processes. For this purpose, the logistical issues of the WWTPs of each of the 3 pre-selected waste water types (irrespective of the type of technology) have been taken into account, with WWTP 3 being the most favourable waste water (as explained in sections 3.1.1 y 3.2.1).

Table 3-2. Analysis of environmental aspects. Table 3-2 shows the main conclusions of the environmental aspects with (-) environmentally unfavourable parameter and (+) environmentally favourable parameter.

Table 3-2. Analysis of environmental aspects.

	Microalgae			MFC		
	WWTP 3	WWTP 4	WWTP 5	WWTP 3	WWTP 4	WWTP 5
Expendable consumption	+	+	+	-	-	-
Energy consumption	-	-	-	+	+	+
Costs	+	+	+	-	-	-
Transport	+	-	-	+	-	-



3.2.7 Microbiological processes WalLAB assessment

Considering all the above-mentioned analysis a final summary table has been created to provide a clear scientific based pilot facility and technology selection procedure. Table 3-3 shows the main conclusions taking into account all the analysed parameters.

Table 3-3. Microbiological process WalLAB assessment.

	Microalgae			MFC		
	WWTP 3	WWTP 4	WWTP 5	WWTP 3	WWTP 4	WWTP 5
Nutrient Recovery Yield	+	+	+	-	-	-
COD in the effluent	+	+	+	- (above legal limits)	- (above legal limits)	- (above legal limits)
Operation time	3 days	3 days	3 days	7 days	7 days	7 days
Environmental aspects	++	0	0	0	--	--

3.3 Next steps

According to the results presented in deliverables D2.3 and D2.8, the main operating parameters, of two nutrient recovery technologies based on microbiological processes, have been studied and selected in order to design and implement one of these technologies at pilot scale. Finally, the selected technology will be the treatment by means of microalgae growth. In contrast, the MFC technology, although it is expected to have a good projection in the future, still needs to continue generating more knowledge on a smaller scale in order to be able to be scaled up.

On the other hand, it has also been technically, economically and environmentally demonstrated that the waste water to be used as raw material for the pilot plant will come from WWTP 3. Therefore, the pilot plant will be installed in Central-Western Spain in accordance with the location of WWTP 3.

The preliminary design will follow the configuration included in Figure 3-3, with a scale-up treating capacity of 500 L WW/day.

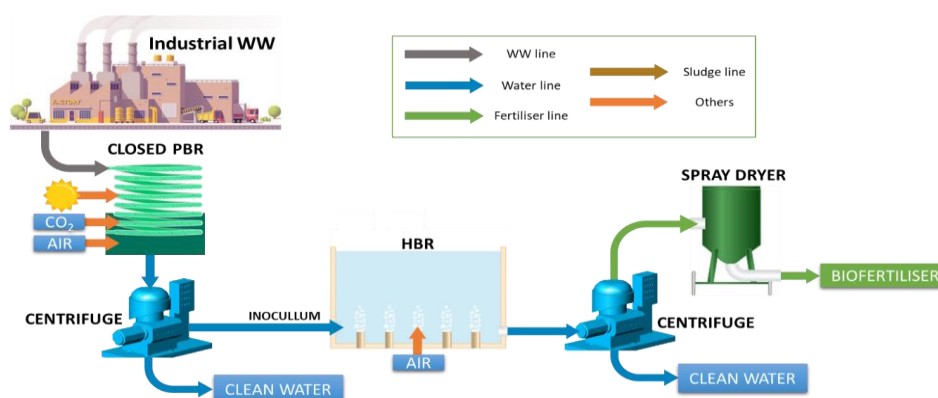


Figure 3-3. Pre-design of the process to be implemented in Pilot 1.

3.4 Conclusions

The results presented in this section provide the following conclusions:



- The Pilot 1 will be located in WWTP 3 due to, the better nutritional composition and the better results obtained at laboratory scale with respect to the other waste water types from the other WWTPs initially considered. Additionally, but not as a determining factor, the distance to the CARTIF and VEOLIA facilities has been also considered.
- Hybrid system of photoautotrophic and heterotrophic microalgal cultivation (microalgae treatment technology) is the microbiological process selected for implementation at pilot scale.



4. HRAS/CS + adsorption/ion-exchange

4.1 High-rate contact stabilisation and adsorption

UGENT and Aquafin tested a 2-stage nutrient recovery system to recover NH_4^+ from municipal waste water (Figure 4-1). The first stage consisted of a high-rate activated sludge process in contact-stabilisation mode (HRAS/CS). The aim of this stage is to remove the organic matter, biological oxygen demand (BOD) as well as chemical oxygen demand (COD), from the municipal waste water and capture it in sludge, while only partially removing total nitrogen. This is achieved by applying a high organic loading rate (OLR) and low sludge retention time (SRT) to the system and providing it with air to activate microbial activity. The contact-stabilisation mode of operation is applied to promote the biosorption of colloidal organics that do not settle by themselves. The contact-stabilisation mode consists in aerating settled (return) sludge in a stabilisation phase to promote biomass growth on stored and adsorbed substrates. Subsequently, the sludge is mixed with influent in a non-aerated contact phase to allow besides regular growth, adsorption and storage of new substrates (present in the influent). This creates a feast-famine cycle in which the aeration of the sludge allows regeneration of its sorption and storage capacity (Meerburg et al., 2015). This stage was operated in pilot-scale, with a sequencing batch reactor (SBR) with a working volume of 378 L.

The aim of the second stage is to adsorb and up-concentrate the ammonium nitrogen in the effluent from the HRAS/CS system on a natural adsorbent or a synthetic resin. The NH_4^+ is adsorbed via ion exchange and physical/chemical adsorption processes (Alshameri et al., 2014; Asano et al., 2007). The regeneration solution and/or the natural adsorbents saturated with ammonium can then be tested as bio-based fertilisers on soil and crops. This stage was carried out in the laboratory scale in 20 x 1 cm (height x diameter) burettes.

The HRAS/CS waste water treatment process allows for a downstream recovery of nitrogen via the adsorption of ammonium cations. The integration of these two processes is proposed as an alternative to the conventional activated sludge process (CAS) (with nitrification and denitrification), where nitrogen is not recovered, but oxidised, using a large amount of energy for aeration. The oxidised nitrogen is then lost to the atmosphere as N_2 and as the greenhouse gas N_2O . Furthermore, the HRAS/CS process has been found to produce sludge with a higher biomethane potential than CAS (Bolzonella et al., 2005). Thus, a HRAS/CS system has the potential to reduce the energy footprint of WWTPs by using less aeration and producing more biogas energy. Therefore, a nutrient recovery system integrating HRAS/CS with adsorption could allow the recovery and reinjection of nitrogen into the economy, as well as decrease the energy footprint and N_2O emissions of waste water treatment plants (WWTPs).

4.2 Alternatives: Description and technical comparison

Alternatives were considered within both stages of the nutrient recovery system (Figure 4-1). For stage 1, different feedstocks were considered for the SBR (Section 4.2.1). The feedstock employed in the first stage influences the capability of the microbiology in the system to metabolise organic matter and reduce the waste water's COD. For stage 2, different adsorbents and resins were tested, in order to choose the best packing material for the adsorption column for bio-based fertiliser (BBF) production (Section 4.2.2). Additionally, 3 different N-recovery methods were tested in the adsorption stage: 1 adsorption cycle without regeneration, adsorption with regeneration and adsorption with regeneration followed by a second adsorption cycle (Section 4.2.3). In these sections, the alternatives considered for each stage will be described and compared from a technical point of view.



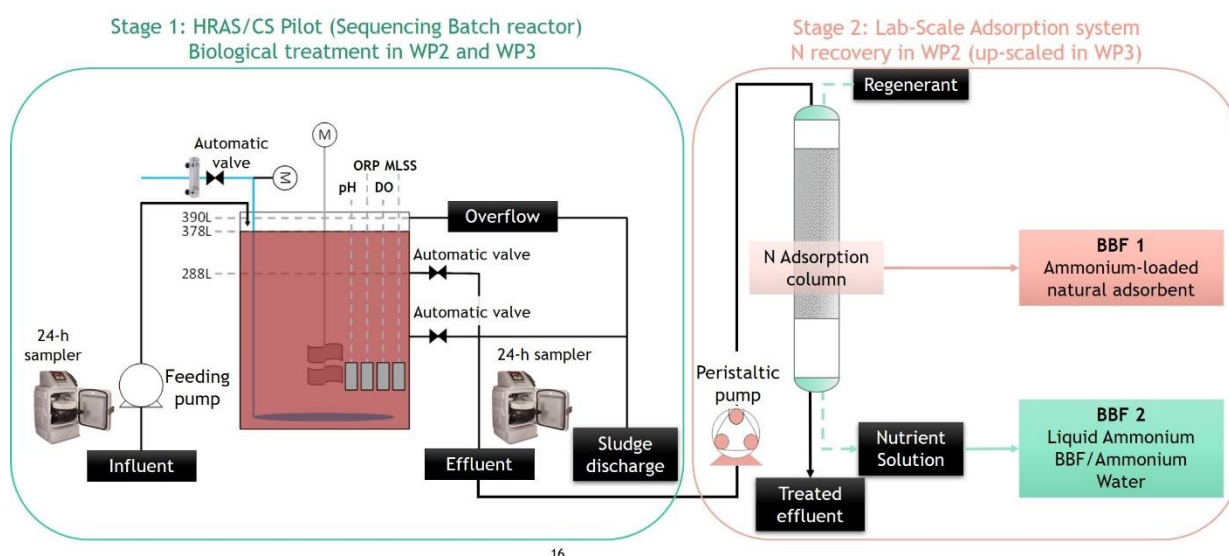


Figure 4-1. Scheme of the proposed NH₄⁺-N recovery system and products. Stage 1: High rate activated sludge with contact stabilisation (HRAS/CS); stage 2: Adsorption column producing bio-based fertilisers (BBFs).

4.2.1 Stage 1: HRAS/CS feedstocks

The waste water influent data for the year 2022 of 5 different full-scale WWTPs operated by Aquafin (Aartselaar, Antwerpen-Zuid, Deurne, Gent and Genk) were studied and compared. Table 4-1 shows an overview of the most relevant physicochemical parameters of the waste water treated at these WWTPs.

The influents were compared by evaluating their yearly weighted average (n=24) and the minimum and maximum daily concentrations for the most representative parameters (biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SS), NH₄⁺-N, total Kjeldahl nitrogen (TKN) and PO₄-P). It was concluded that the difference between the averages of these parameters were not highly significant. Therefore, Aquafin and UGent decided to only use the influent of the WWTP of Aartselaar for this task because of (1) its representativeness for most other installations of Aquafin and (2) its BOD/N ratio of 3.4 is suitable to apply during this task.

4.2.2 Stage 2: Natural adsorbents and synthetic resins

Five different materials were tested for their ammonium adsorption efficiency: two commercially available synthetic resins (Purolite c100h and Amberlite irc120h) and three commercially available natural adsorbents (zeolites, Biochar 1 and Biochar 2). Zeolites were tested in two ionic forms (Na⁺ and H⁺) by washing the zeolites and then incubating them overnight, in 2 batches, with either 13,4% (w/v) NaCl (for Na⁺ form) or 1.5% (w/w) HCl (for H⁺ form). Table 4-2 shows important characteristic of the five tested materials, including ionic form, particle size and cost.

Table 4-1. Characteristics of waste water (yearly weighted average [n=24]) treated at different WWTPs (Aartselaar, Antwerpen-Zuid, Deurne, Gent and Genk) operated by Aquafin. Minimum and maximum daily concentrations are shown in parentheses. All results are based.

Parameters	Waste water Treatment Plants				
	Aartselaar (54.000 PE)	Antwerpen- Zuid (171.000 PE)	Deurne (193.500 PE)	Gent (207.000 PE)	Genk (63.000 PE)
BOD (mg.L ⁻¹)	93 (27-290)	130 (65-230)	136 (53-260)	82 (32-150)	83 (28-160)
COD (mg.L ⁻¹)	316 (120-690)	406 (230-710)	389 (250-690)	259 (130-570)	285 (140-970)



Suspended solids (mg.L ⁻¹)	129 (62-360)	163 (87-330)	164 (86-390)	116 (72-410)	131 (48-290)
NH ₄ -N (mg.L ⁻¹)	28 (6.2-59)	38 (15-70)	38 (15-74)	34 (13-65)	18 (8.0-36)
TKN (mg.L ⁻¹)	35.5 (7.3-69)	47.0 (20-78)	45.6 (24-78)	38.6 (17-70)	25.5 (18-45)
PO ₄ -P (mg.L ⁻¹)	2.9 (0.6-6.6)	3.5 (3.0-9.9)	3.3 (1.3-6.9)	3.6 (1.2-9.0)	1.6 (0.5-3.1)

These materials were tested in batch experiments. One set of batch experiments was carried out to determine the ammonium nitrogen removal efficiency and adsorption capacity of each material, as well as to plot their adsorption isotherms. The experiments consisted in adding seven different dosages of the adsorption materials (1.0, 2.5, 5.0, 7.5, 10.0, 12.5 and 15.0 g/L) in a 25.0 ± 1.5 mg/L NH₄⁺-N solution. Figure 4-2 describes the experimental approach used to obtain removal efficiency and adsorption capacity data for each material.

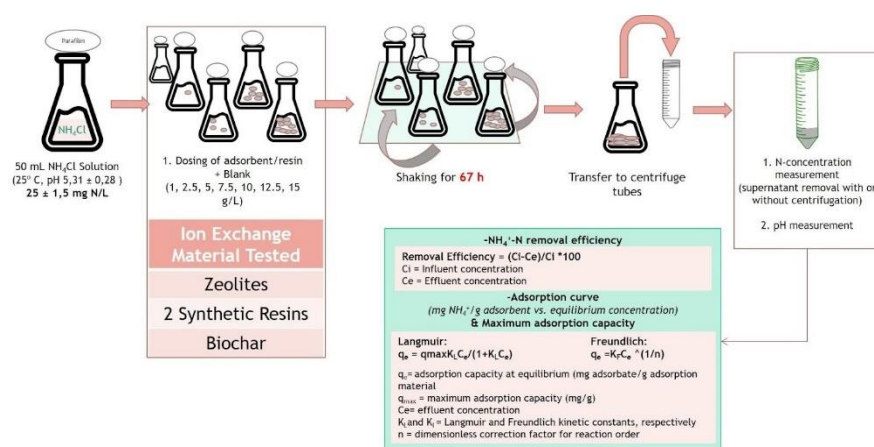


Figure 4-2. Experimental set-up of the batch experiment run to plot the adsorption isotherms of the different adsorption materials tested and to determine their ammonium removal efficiency and adsorption capacity.

The zeolites in Na⁺ form (Z-Na), Purolite c100h (PL) and Amberlite irc 120h (AL) achieved the highest removal efficiencies, reaching >85% for dosages between 2.5-15.0 g adsorbent/L. The zeolites in H⁺ form (Z-H) performed less efficiently than Z-Na, but still reached a removal efficiency >85% at dosages of 7.5-15.0 g Z-H/L. The Biochar 1 (B1) did not achieve a removal efficiency larger than 30 %, and Biochar 2 (B2) did not manage to adsorb any NH₄⁺-N at the tested dosage of 2.5 g B2/L, with a final equilibrium concentration (C_e) equal to the initial concentration (C_i). Figure 4-3 shows the average removal efficiency of PL, AL, Z-Na, Z-H and B1 at all dosages tested.

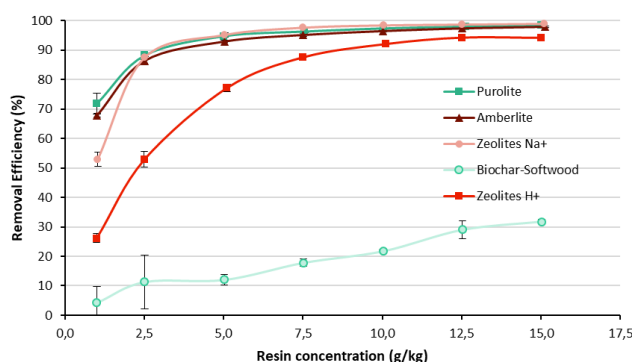


Figure 4-3. Average ammonium nitrogen removal efficiency of five of the adsorbent materials. Standard deviations shown in bar format.

The adsorption isotherms of each adsorbent were modelled with the Langmuir and Freundlich models, when possible. The equations used to model these adsorption isotherms are shown in Figure 4-2. Table 4-3 shows the Langmuir and Freundlich constants calculated for PL, AL, Z-H and Z-Na. PL and AL adsorbents had the best fit with the Freundlich model, therefore the maximum adsorption capacity (q_{\max}) cannot be estimated from the calculated kinetic parameters. The Z-Na⁺ adsorbent best fit the Langmuir model, with a q_{\max} of 13.61 mg N/g. The Z-H⁺ adsorbent fit both models well, with a q_{\max} of about half that of Z-Na⁺ ($q_{\max} = 7.54$ mg N/g). It was not possible to model the adsorption isotherm of B1 due to its poor adsorption capacity at the tested ammonium loads. The PL adsorption isotherm showed the best (i.e., highest) ratio of adsorption capacity relative to the final ammonium concentration in the solution, followed by AL (Figure 4-4).

Table 4-2. Materials tested for their ammonium adsorption efficiency.

Adsorbent/ Resin	Type & Ionic Form	Particle Size (μm)	Cost
Purolite c100h	Strong acid cation resin – H ⁺ form	300-1200	€ 2990/m ³ (for orders > 2 m ³) € 4.08/kg (for 120 kg orders)
Amberlite irc 120h	Strong acid cation resin – H ⁺ form	620-830	€ 35/kg (for 100 kg orders)
Zeolites	Natural aluminosilicate mineral – Na ⁺ and H ⁺ form	50-1000	€ 150/m ³ € 0.147/kg
Biochar 1	Mixed hardwood (80%) and softwood (20%) (Carbon Terra) – Na ⁺ form	500-1000	Data not available
Biochar 2	Garden waste (GreenPoch) – H ⁺ form	< 3000	€ 869.69/m ³

Another set of kinetic batch experiments were carried out to determine how quickly the ammonium adsorption reaction takes place on the different materials. The experiment consisted in sampling a 25.0 ± 1.5 mg/L NH₄⁺-N solution containing 2.5 g/L of adsorption material at 5 different time points: 0.58, 1.5, 3, 24 and 45 hours. This concentration of the adsorbent material was chosen based on removal efficiency results from the adsorption batch experiment (Figure 4-3).

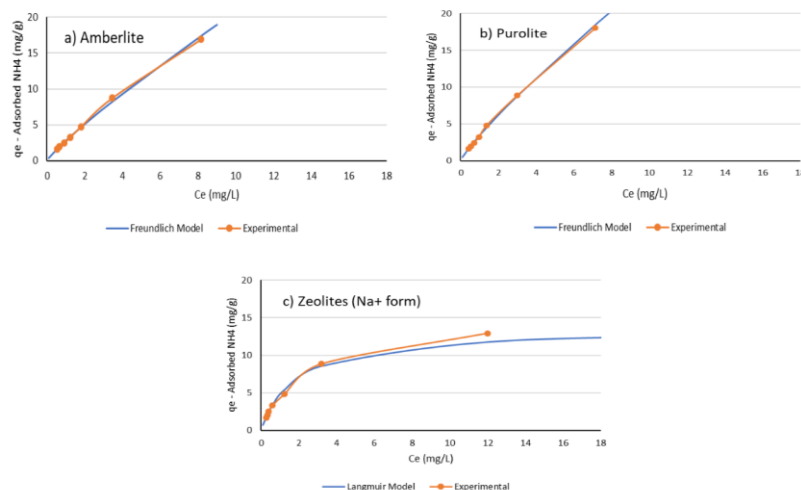


Figure 4-4. Adsorption isotherms of a) Amberlite irc120h, b) Purolite c100h and c) zeolites in Na⁺ form. Only the models (blue lines) that best fit the experimental data are shown (orange dotted lines).

Data from the kinetic tests showed that the adsorption kinetics of PL, AL and Z-Na were fast. The adsorbed NH₄⁺-N on PL and AL adsorbents reached equilibrium after 1.5 h, and on Z-Na after 3 h. Average equilibrium concentrations for PL, AL and Z-Na were 8.8, 8.9 and 8.7 mg/g, respectively. For B1 and B2, no significant adsorption was observed at this dosage (2.5 g/L), even after 45 h. Figure 4-5 shows kinetic experimental data for PL, AL and Z-Na.

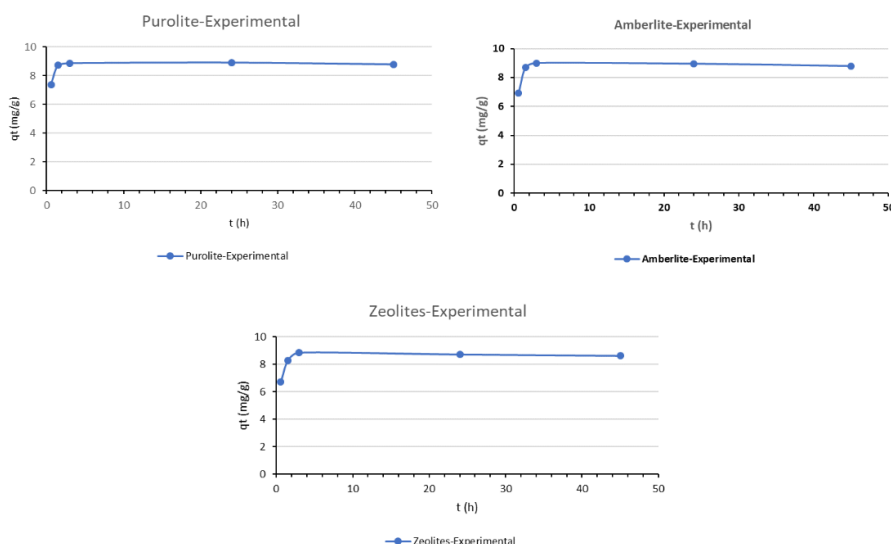


Table 4-3. Langmuir and Freundlich adsorption modelling for tested adsorption materials.

Adsorbent/Resin	Langmuir			Freundlich		
	q_{\max} (mg/g)	K_L (L/g)	R	n (-)	K_F (g/g)	R
Purolite c100h	34.01	0.12	0.9967	1.16	3.41	0.9985
Amberlite irc120h	29.24	0.11	0.9935	1.15	2.78	0.9982
Zeolites (Na ⁺ form)	13.61	0.53	0.9956	1.84	3.94	0.9708
Zeolites (H ⁺ form)	7.54	0.21	0.9888	1.97	1.53	0.9975

The kinetic experiments were carried out with all materials in Table 4-2, except the Zeolites in H⁺ form. The adsorption batch experiment was carried out with all materials except Biochar 2. The reason for this is that it was determined that enough data was collected from the rest of the batch experiments, in order to make a decision regarding the packing material to be used for the laboratory-scale column tests.

Even though the synthetic resins showed the best adsorption efficiency (based on the adsorption isotherm batch experiments), as well as fast kinetics, zeolites were the chosen adsorbent for further laboratory scale testing.

Figure 4-5. Kinetic data for Purolite c100h, Amberlite irc120h and zeolites in Na⁺ form.

The reasons for these include (i) that choosing a natural adsorbent would allow the testing of both the saturated adsorbent and spent regenerant as BBFs, which better fulfills the project objectives, (ii) Z-Na also showed fast adsorption kinetics, (iii) NH₄⁺ removal efficiency of Z-Na was comparable to PL and AL, and Z-H achieved high removal efficiencies at high dosages, unlike biochar and (iv) zeolites showed the best life cycle sustainability (see Section 4.2.4.2).

4.2.3 Stage 2: NH₄⁺-N Recovery method

Three different recovery methods were tested in the laboratory scale in 20 x 1 cm (height x diameter) burettes packed with zeolites (chosen based on results from the technical and LCSA analyses in Sections 4.2.2 and 4.2.4.2, respectively). The three N recovery methods tested were the following:

1. 1 adsorption cycle followed by the harvest of ammonium-loaded zeolites (1A).
2. 1 adsorption cycle followed by one regeneration cycle and harvest of spent regenerant (A-R).



3. 1 adsorption and regeneration cycle (with harvesting of spent regenerant) followed by a second adsorption cycle and the harvest of ammonium-loaded zeolites (A-R-A).

The columns were packed with 10.2 g of zeolites (equal to a bed volume of 10 mL). The zeolites were used in their original ionic state (Z-O). Synthetic waste water (Table 4-4) was used during the adsorption cycle to saturate the zeolites, and a 3% (w/w) KCl solution (pH = 5.09) was used as regenerant during the regeneration cycle. Both solutions were run through the columns at a flow rate of 40 bed volumes (BV) per hour. One BV is equal to the empty bed volume of a packed column (i.e., for a packed bed of 1 L, 10 BV is equal to 10 L). Figure 4-6 depicts the set-up used for these column tests.

The adsorption cycle was defined as the saturation of zeolites with $\text{NH}_4^+\text{-N}$ until the cumulative effluent concentration of $\text{NH}_4^+\text{-N}$ reached the discharge limits (20% of the initial concentration). In this experiment, a full adsorption cycle treated 140 BV of synthetic waste water. The (concurrent) regeneration cycle was defined as the desorption of $\text{NH}_4^+\text{-N}$ from zeolites until a stable, low $\text{NH}_4^+\text{-N}$ concentration is reached in the discrete effluent samples. In this experiment, a full regeneration cycle was completed after 40 BV. The liquid BBF obtained after regeneration was defined to be the spent regenerant collected up to 1 BV of outflow (i.e., only the first 10 mL of effluent obtained from the columns were considered as liquid BBF). The remaining 39 bed volumes were discarded.

Table 4-4. Salt content and ionic concentrations of the synthetic waste water used to test different N recovery methods from adsorption columns

Salt Content	Cation Concentration (mg/L)
NH_4Cl	25
NaCl	103.7
$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	79.5
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	7.9
KH_2PO_4	25.4

In the adsorption cycles, the cumulative $\text{NH}_4^+\text{-N}$ concentration was measured in the effluent reservoir and in the regeneration cycle $\text{NH}_4^+\text{-N}$ was measured both in the effluent reservoir (cumulative concentration) and in discrete effluent samples. The concentration of ammonium adsorbed on the saturated zeolites was also determined by extracting the $\text{NH}_4^+\text{-N}$ in a 1 M KCl solution (in a 1:10 ratio [zeolites to KCl solution], shaken for 1 h at 150 rpm). Spectrophotometry test kits from the Nanocolor brand were used to carry out the $\text{NH}_4^+\text{-N}$ measurements in solution.

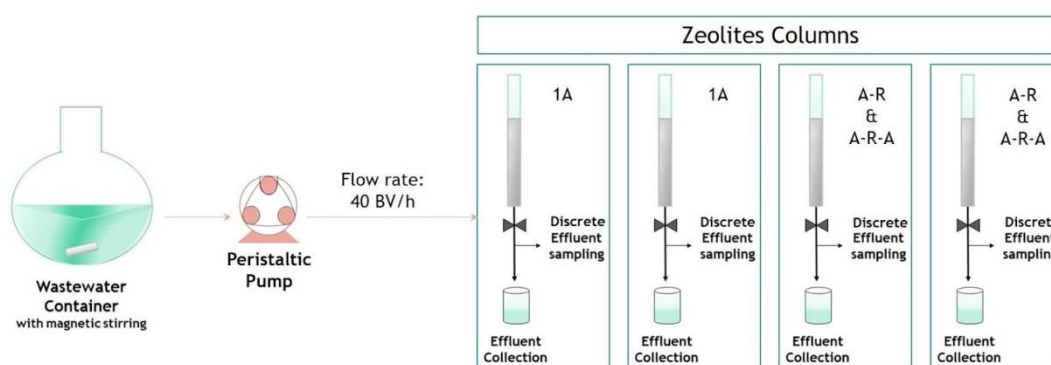


Figure 4-6. Set up of the experiments testing of three N recovery methods (1A, A-R and A-R-A). 1A: 1 adsorption cycle with N recovery as ammonium-saturated zeolites (1A). A-R: an adsorption cycle followed by a regeneration cycle, in which N is only recovered in 1 BV of spent regenerant. A-R-A: A second adsorption cycle (following the N recovery in spent regenerant described in A-R) in which N is recovered as ammonium-loaded zeolites.

The description and results of the different nitrogen recovery methods is summarised in Table 4-5. The quantity of product obtained per BV of waste water treated is the same for all three tested recovery methods (Figure 4-2). However, the $\text{NH}_4^+\text{-N}$ obtained per BV of waste water treated with the method 1A (0.0135 g/kg·BV) is



larger than that obtained with A-R and A-R-A (0.0071 and 0.0097 g/kg·BV, respectively). It is predicted that when scaling up the column to the pilot nutrient-recovery system, the product obtained will also be equal to around 0.0071 bed volumes/BV waste water treated for all recovery methods. Therefore, from a technical point of view, method 1A is the best in terms of N recovery.

These tests also showed that Z-O had a treatment capacity of 140 bed volumes of waste water with an $\text{NH}_4^+\text{-N}$ removal efficiency of 80% in the cumulative effluent ($C_i = 24.4 \text{ mg/L NH}_4^+\text{-N}$) and a total $\text{NH}_4^+\text{-N}$ recovery efficiency of 57%, for the first adsorption cycle. The $\text{NH}_4^+\text{-N}$ recovery efficiency from A-R was 29% (140 BV treated) and from A-R-A, 40% (280 BV treated). However, for A-R-A, the removal efficiency dropped to 73% on the second adsorption cycle. These results show that zeolites in their original ionic state can achieve a satisfactory ammonium removal efficiency.

Table 4-5. Description and results of different nitrogen recovery methods. BV = bed volumes.

	Description	Recovered product	$\text{NH}_4^+\text{-N}$ concentration	Product quantity (Bed volumes product/BV waste water treated)	N quantity (g/kg·BV waste water treated)
1 Adsorption cycle (1A)	One adsorption cycle ^a	Solid product (ammonium-saturated zeolites)	$1.9 \pm 0.1 \text{ g/kg}$	0.0071	0.0135
Adsorption-Regeneration (A-R)	Adsorption cycle ^a followed by a regeneration cycle ^b	Liquid product (ammonium-saturated spent regenerant)	Liquid product: $1.00 \pm 0.04 \text{ g/L}$	0.0071	0.0071
Adsorption-Regeneration-Adsorption (A-R-A)	Adsorption cycle ^a followed by a regeneration cycle ^b , followed by a 2 nd adsorption cycle ^a	Solid (ammonium-saturated zeolites) and liquid product (ammonium-saturated spent regenerant)	Solid product: $1.7 \pm 0.0 \text{ g/kg}$	0.0035 (solid)	0.0061 (solid product)
			Liquid product: $1.00 \pm 0.04 \text{ g/L}$	0.0035 (liquid)	0.0036 (liquid product)
				0.0071 (total)	
					0.0097 (total)

^a An adsorption cycle is defined as the saturation of a zeolites with $\text{NH}_4^+\text{-N}$ until the cumulative effluent concentration of $\text{NH}_4^+\text{-N}$ reaches discharge limits. These adsorption cycles treated 140 BV of synthetic waste water

^b A regeneration cycle is defined as the desorption of $\text{NH}_4^+\text{-N}$ from zeolites until a stable, low $\text{NH}_4^+\text{-N}$ concentration is reached in the discrete effluent samples. A full regeneration cycle was completed after 40 BV.

4.2.4 Life Cycle Sustainability comparison

In this section, the alternatives presented for stage 1 [HRAS/CS] and stage 2 [adsorption] of the proposed nutrient recovery system (Figure 4-1) will be compared from a Life Cycle Sustainability Assessment (LCSA) perspective.

4.2.4.1 Stage 1: HRAS/CS feedstocks

All feedstocks considered for HRAS/CS treatment would generate a similar impact per population equivalent (PE) from an LCSA perspective. The energy consumption per PE related to the removal of organic matter and



the cost per PE related to removal of organic matter and recovery of nitrogen would be similar given the concentration of these components is similar between the feedstocks.

4.2.4.2 Stage 2: Natural adsorbents and synthetic resins

For the LCSA comparison, the adsorption materials tested in WP2 (Table 4-2) can be compared based on energy consumption, cost, transport and waste generation. The production of all tested materials entails the use of energy, namely energy related to the production of synthetic resins and biochar and energy related to the mining of zeolites. However, a comparison on the basis of energy consumption was not possible, given information on the energy consumption for the production or mining of the adsorption materials is not readily available. The impact from the cost of the materials was considered based on the bulk costs shown in Table 4-2. Zeolites were the least costly, followed by Biochar 2 (B2). Biochar 1 (B1) was not accounted for in this comparison because it was donated by another research group in Gent University. A price quotation was not requested and further investigation about whether bulk quantities of B1 could be provided was not carried out given the test results obtained for this biochar (<35% removal efficiency) already showed it was not appropriate for further testing. The Amberlite irc 120h (AL) synthetic resin was the most costly adsorption material tested.

Regarding transport, the distance of transportation of the product to the Aquafin Research Hall was taken into account. The Purolite c100h (PL) synthetic resin is manufactured and delivered from Romania; AL synthetic resin, from the United States; B1, from Germany; B2 from Belgium and the zeolites are mined in and delivered from Slovakia. Therefore, from a transport point of view, B1 and B2 imply the least negative impact, followed by zeolites and PL. AL use implies the largest impact from transport. Finally, the use of synthetic resins would generate more waste, given that they would need to be discarded after a few cycles of use. On the other hand, the zeolites and biochar could be potentially reused as bio-based fertilisers, and would not be wasted. Table 4-6 compiles the comparison of the different adsorption materials on an LCSA basis. From these analyses, the zeolites and B2 present the highest life cycle sustainability.

Table 4-6. Comparison of different feedstocks for the high rate activated sludge treatment with contact stabilisation from a life cycle sustainability assessment perspective.

	WWTP				
	Aartselaar (54,000 PE)	Antwerpen- Zuid (171,000 PE)	Deurne (193,500 PE)	Gent (207,000 PE)	Genk (63,000 PE)
Energy consumption	=	=	=	=	=
Cost	=	=	=	=	=

Table 4-7. Comparison of different adsorption materials from a life cycle sustainability assessment perspective.

	Adsorption Material				
	Purolite c100h	Amberlite irc 120h	Zeolites	Biochar 1	Biochar 2
Cost	++	+++	-	N/A	+
Transport	++	+++	++	+	-
Waste generation	+	+	-	-	-

4.2.4.3 Stage 2: N Recovery Method

From the LCSA perspective, the N recovery methods tested (1A, A-R and A-R-A, described in Table 4-5) were compared based on energy and reagent consumption, space requirements, cost and waste generation. Bed volumes (BV) were used as the volume unit for reagents and waste water to analyse the experimental data, given this facilitates the extrapolation of data from the laboratory scale (bed volume = 10 mL) to the pilot scale (bed volume >>>10 mL, to be determined for pilot design). One BV is equal to the empty bed volume of a packed column (i.e., for a packed bed of 1 L, 10 BV is equal to 10 L). Energy consumption was analysed qualitatively, based on the amount of NH_4^+ -N recovered per time of operation. The type of energy used for all the recovery methods is electrical energy, needed to power the peristaltic pump. Thus, the longer the operation



time, the higher the energy use. The 1A method achieved the highest N recovery in the shortest amount of time (0.54 g $\text{NH}_4^+\text{-N/kg}$ zeolites·hour), meaning that it requires the least amount of pumping for recovering a given amount of $\text{NH}_4^+\text{-N}$ mass. The A-R and A-R-A methods recovered 0.28 g $\text{NH}_4^+\text{-N/kg}$ zeolites·hour and 0.39 g $\text{NH}_4^+\text{-N/kg}$ zeolites·hour, respectively.

Reagent consumption is highest for A-R, with 4.0 BV of 3% KCl and 0.1 BV of zeolites needed per mg of N recovered, for a total of 4.1 BV of input per mg N recovered. A-R-A reagent consumption is the second highest, with a total input of 1.54 BV/mg N recovered (1.5 BV of 3% KCl and 0.04 BV of zeolites per mg N recovered); and 1A consumes the least materials, with 0.05 BV of zeolites per mg N recovered. In terms of the cost of materials, the cost of KCl (€0.15, [Alibaba](#)) and zeolites (€0.147/g, from Table 4-2) were taken into account. Considering a packed bed volume of 1 L containing 1.0 kg of zeolites, 1A is the least expensive method, costing €0.15 per full N recovery cycle. A-R and A-R-A would incur a cost of €0.33/cycle linked to the use of 1 kg of zeolites and 40 BV of a 3% KCl solution as regenerant. As 1A also recovers the most N per cycle, it is also the least expensive in terms of N recovery (followed by A-R-A).

Regarding space requirements, A-R and A-R-A recovery methods would require more space due to the need of a regeneration storage tank and a liquid BBF storage tank. Finally, when using the A-R or A-R-A nitrogen recovery method, a fraction of spent regenerant must be discarded, given the cumulative concentration of $\text{NH}_4^+\text{-N}$ decreases significantly after 1 BV of spent regenerant is collected. This means that after collecting 1 BV of spent regenerant as BBF, the remaining 39 BV of spent regenerant must be treated as waste. If using the A-R nitrogen recovery method, the zeolites would also need to be discarded. Therefore, A-R and A-R-A would generate a larger amount of waste than 1A. Table 4-7 compiles the comparison of the different N recovery methods on an LCSA basis.

Table 4-8. Comparison of different N recovery methods from a LCSA perspective. All sustainability parameters are considered based on N recovery within a full cycle.

	NH ₄ ⁺ -N Recovery method		
	1 Adsorption Cycle	Adsorption-Regeneration	Adsorption-Regeneration-Adsorption
Energy consumption	-	++	+
Reagent consumption	-	++	+
Space requirements	-	+	+
Cost	-	+	+
Waste generation	-	++	+

4.3 Next steps

The results presented in this deliverable were used to make final design decisions for the pilot-scale nutrient recovery system (Figure 4-1) that will be tested in WP3. For the 1st stage of the system (HRAS/CS), the waste water collected at the Aartselaar WWTP site was chosen as feedstock for the HRAS/CS SBR.

For the 2nd stage (adsorption/ion exchange), two main design decisions were made based on the results presented and comparisons made in the previous section:

1. Zeolites in their original ionic state (Z-O) were chosen as the column packing material for laboratory column tests and WP3.
2. The N recovery methods 1A and A-R-A (Table 4-5) were chosen as recovery methods to be optimised in WP3.

Zeolites were chosen as packing material for the pilot-scale column, and they were also used for the laboratory-scale column tests, after comparison with other adsorbent materials (Table 4-2 and Table 4-7). The main reasons for choosing zeolites were (1) their potential for high $\text{NH}_4^+\text{-N}$ removal efficiency from an ammonium salt solution as both Z-Na and Z-H (Figure 4-2), (2) the fact that they allowed the testing of 3 different N recovery methods (Table 4-5) and (3) their higher life cycle sustainability compared to other materials (least expensive, proximity of source and potentially lower waste generation). It was decided to use zeolites in their original ionic state, rather than zeolites in Na^+ form, in order to simplify the adsorption process by reducing reagent consumption and pre-processing steps. Z-O was tested in multiple laboratory-scale column



experiments and proved that it could be used successfully to remove and recover $\text{NH}_4^+\text{-N}$ from a satisfactory quantity of bed volumes of waste water.

The 1A and A-R-A were chosen as N recovery methods after consideration of technical, LCSA and practical aspects. From a technical and LCSA points of view (Table 4-5 and Table 4-8, respectively), the 1A recovery method showed the best performance. However, after internal meetings between Aquafin and UGent, the following was concluded:

- A-R-A creates more opportunities for optimisation of the adsorption pilot: 1) optimisation of the saturation cycles, to increase N recovery from both the regenerant and final adsorbent and 2) optimisation of the regeneration cycle to decrease the amount of regeneration waste generated.
- The regeneration effluent produced in A-R-A (with KCl regenerant) can be tested as K fertiliser or irrigation water. The production of irrigation water could be of value if produced in an on-farm system to replace other irrigation sources or as back-up during drought periods.
- The operation of the pilot nutrient recovery system is more feasible with the A-R-A method, given it is easier for Aquafin to manage liquid regeneration waste than solid zeolites waste. The quantity of zeolites BBF produced during WP3 will considerably surpass the amount of BBF needed to carry out pot experiments during WP4 but will not generate enough BBF to test in the field.

Therefore, during WP3, both 1A and A-R-A methods will be optimised. Saturated zeolites will be produced with both the 1A and A-R-A methods (to be tested in WP4) and the A-R-A method will be applied and optimised during operation periods in which it is not necessary to harvest BBFs for pot trials.

4.4 Conclusions

Different scenarios were considered for both the first (HRAS/CS) and second stages (adsorption) of the proposed nutrient recovery system (Figure 4-1). For the first stage (HRAS/CS), different feedstocks collected at different WWTPs operated by Aquafin were considered. The existing data from the year 2022 for representative parameters (COD, BOD, SS, $\text{NH}_4^+\text{-N}$, TKN and $\text{PO}_4\text{-P}$) were assessed to determine the suitability of the different collected waste waters for use as feedstock for the HRAS/CS sequencing batch reactor. It was concluded that the collected waste waters did not differ significantly from each other. Therefore, the waste water collected at the Aartselaar WWTP site was chosen to carry out the pilot HRAS/CS process, due to its representativeness of the other collected streams and to its appropriate BOD/ $\text{NH}_4^+\text{-N}$ ratio of 3.4.

For the second stage (adsorption), five different adsorption materials (Table 4-2) and three different N recovery methods (Table 4-5) were tested and compared to each other on a technical and LCSA perspective. After consideration of technical, sustainability and practical aspects, it was decided to use zeolites in their original ionic state (Z-O) and the 1-A and A-R-A N-recovery methods for the pilot-scale adsorption system. When tested in a column for synthetic waste water treatment, Z-O achieved a treatment capacity of 140 bed volumes (BV) of waste water with an $\text{NH}_4^+\text{-N}$ removal efficiency of 80% in the cumulative effluent ($C_i = 24,4 \text{ mg/L NH}_4^+\text{-N}$) and an $\text{NH}_4^+\text{-N}$ recovery efficiency of 57% after the first adsorption cycle. Using the A-R-A nitrogen recovery method, the N recovery efficiency dropped to around 40%. However, with the A-R-A method, more BVs of waste water can be treated with a reduced production of solid BBF per recovery cycle. This facilitates the management of products obtained from the nutrient recovery system in WP3. A-R-A also allows for the optimisation of both saturation and regeneration cycles and expands the potential target group the proposed nutrient recovery system to farms (in addition to WWTPs).



5. ABC-BioPhosphate: multi-functional biofertiliser and adsorbent

5.1 Brief description of the aim of the 3R technology/pilot 3 and main characteristics

The WalNUT_3R_TRL5 pilot plant no.3 demo is aiming to provide solution for unexploited dairy food industrial WW stream that is validated in relevant environment while environmental, regulatory and socio-economic issues are defined and qualitatively assessed. The WalLAB circular methodology to scale-up 3R technology to TRL5 pilot level is to design, develop and test a user/market driven upcycling alternative for dairy food industrial WW acidic whey challenges and provide an economically improved and environmentally sustainable recovery solution for the sector, in line with the objectives of the EIP on Agrifood and Raw Materials. In pilot plant 3 dairy acidic whey food industrial WW is upcycled in combination with other food industrial by-products, such food grade bone meal processed (ABC-animal bone char), tomato pulp by-products and composted animal manure. The objective is the integrated up-cycling of unexploited food industrial WWs and by-products towards systems with scale of economy, improve agri-resource management for less cost and recycle essential nutrients back into the soil. The aim of the 3R - WP2 is to design, develop and test sustainable and innovative technology at TRL5 for dairy industrial nutrient recovery from acidic whey waste water raw material streams. High nutrient density biofertiliser production is targeted in combination with recovered phosphorus from animal bone char adsorbent, as part of STO2, implemented and lawfully operated in the WP3. The output, high nutrient density biofertiliser products, is expected to be delivered to local farmer users to close the nutrient loops, and they will use it for their onsite CMC3 compost material nutrient enrichment and improvement.

The three specific objectives are:

- a) Develop a standard protocol to lawfully characterise dairy industrial acidic whey WW streams and evaluate technology with fungus fermentation process.
- b) Design and test technology for NR at lab scale from dairy WW acidic whey stream as main target.
- c) Evaluate and select the best technology for lawful and relevant environment operated large scale prototype TRL5, aiming upcycling of dairy WW stream for nutrient recovery; while considering technical, economic, environmental and climate aspects that all together can result integrated business model under market competitive conditions.

The 3R provides biotech solution for the nutrient recovery from the main WW stream acidic whey, produced by the dairy food industry that unexploited and problematic WW is converted into a recovered resource raw material and upcycled products. Acidic whey WW is the overwhelming largest and unexploited liquid by-product stream of the dairy food industry with significant economic importance and environmental challenge. This problematic liquid stream is converted into P/Ca based multi-functional compound biofertiliser with combined effects by multi-stage processing for wide range of product functional applications (as of safety, quality and labelling compliance defined by the (EU) 2019/1009 PFC categories) and high nutrient recovery efficiency while environmental impacts minimised. Acidic whey WW will be used as liquid medium during the fermentation process and high concentration biomass of the agriculturally beneficial microbial strain/consortia will be produced that is solid fermented to prepare products to close the nutrient loops for optimised field condition applications. The output product will be aerobic green compost, adapted and possibly combined with vermicomposting as well.

5.2 Description of the alternatives

The challenge of the dairy industrial acidic whey WW with 93% water content is that it is consisting high lactose organic contamination at as low as pH 4.5. This material is perishable and as unstable organic material must be processed within 48 hours, or cooled, in which case must be processed within 14 days. However, due to the very large volume streams, none of the storage alternatives are economically viable in industrial practice. Unlike sweet whey this cannot be utilised directly and therefore providing high treatment cost, while this



problematic WW stream is produced at very large scale. Dairy industrial acidic whey WW, if not properly processed, can cause significant environmental problems; therefore, the lawful utilisation of this stream is a high cost and economic burden for the industry.

Acid whey is a potential hazard to the aquatic environment due to its high organic matter content in the shape of lactose, resulting in a high Biological Oxygen Demand and Chemical Oxygen Demand. The high BOD/COD makes the acid whey WW treatment expensive, particularly in the case of small and medium sized milk processing plants. The high BOD level means that the presence of acid whey in waters would cause a drop in biological oxygen levels, leading to the elimination of aquatic life. Hence, if other uses cannot be found, acid whey must be treated as waste water involving significant financial costs for the dairy as well as socio- economic costs associated with waste treatment. If acidic whey is not processed, it quickly turns sour due to the formation of lactic acid. If its utilisation cannot be resolved, the large amount of acid whey must be separated from the other waste water, because it would result in an unacceptably low pH value for on-site waste water treatment or discharge into the public sewer system. The utilisation of acidic whey is limited due to its different composition from sweet whey. The challenges of utilising acid whey occur in the processing procedure. Spray- drying acid whey with conventional technology is not feasible due to the high content of lactic acid, which makes the whey powder more likely to absorb moisture, resulting in an increased stickiness of the powder. Moreover, a low pH makes the proteins less stable and it is more difficult, for instance, to remove water from acid whey than from sweet whey. Because of the low pH and the proximity to the isoelectric point, the protein will readily precipitate, which may make it difficult to recover.

The usual traditional alternatives to treat dairy industrial acidic whey WW are summarised in Table 5-1.

5.3 Test and studies carried out

In the Task 2.4 the Concept of the “ABC-BioPhosphate: multi-functional biofertiliser and adsorbent” were developed. The liquid/solid state fermentation and integrated ABC-BioPhosphate adsorption technology of food industrial dairy WW stream acidic whey were successfully validated at laboratory scale at petri dish agar culture; 500 ml shake flask cultures and 3x3 litre fermentation for low-cost upcycling and production of recovered BBFs and also the obtainment of high quality final effluent up-cycled to clean water.

Table 5-1. Cost comparison of alternatives for ABC-biphosphate production.

Alternative utilisation list for dairy industrial acidic whey WW	Cost for dairy industry producer
Biogas production at anaerobic digesters to produce methane. However, due to low pH only smaller % of such WW can be added to the biogas production stream.	High gate fee paid by the dairy industry producer to be accepted as anaerobic digester additive.
Nano-filtration processing to extract lactose and milk minerals.	Very high cost for the dairy industry producer and not market competitive utilisation. Highly energy intensive solution.
Land-spread on farmers' fields that is made in the past but now legally restricted as of significant negative environmental impacts.	Illegal action.
Dump to drain that is made in the past but now legally restricted as of significant negative environmental impacts.	Illegal action.
Fungus biotech processing to decompose lactose and organic ingrediency content. The dairy food industrial WW acidic whey will be used as liquid medium during the fermentation process resulting 2 economically high phosphorus content BBF products, such as fungus formulated ABC BBF and nutrient formulated multifunctional adsorbent ABC BBF. Direct adsorption is also an option, but in that case the adsorbent need to be regenerated. The recovered clean water content is used for co-composting irrigation moisture control.	Free take over for lawful upcycling processing of dairy industrial acidic whey WW in large scale.

In this context, upcycling means high added value transformation of unexploited biomass into new products, perceived to be of greater quality and environmental / climate value with second life and new function that finished product becomes more practical and valuable than what it previously was. Upcycling is incl. recycling but at higher level.



b) Adsorption processing of the separated fermentation liquid residuals.

ABC animal bone char was used as adsorber and was compared with coconut shell activated carbon adsorber, both with thermal regeneration opportunity. The low pH is adjusted and neutralised by the ABC high Ca content in combination with food grade sodium bicarbonate water soluble additive (sodium hydrogen carbonate CAS 144-33-8 at pH 8.3). The high BOD, COD and TOC are decreased to meet the permissible limits, but as of thermal regeneration of the spent adsorber the biofertiliser component is also decomposed. Therefore, the fermentation upcycling process is considered as biofertiliser product driven main treatment line and the adsorption processing is a support line to complete the cycle.

Conclusion: successful processing. ABC animal bone char is highly optimal and efficient to adsorb macromolecular organic contamination in liquid stream and adjust pH in combination with food grade sodium bicarbonate. ABC can be thermally regenerated 3-4 times, thereafter, reused as soil biofertiliser as containing economically high phosphorus density. The biofertiliser product driven fermentation upcycling process main treatment line is supported with direct and post processing adsorption liquid treatment options. In this context wide range of flexible and economically viable processing options applied to develop multi-functional biofertilisers. The extracted irrigation clean water is upcycled from the dairy WW stream and used for upgrade of co-composted materials at onsite farm level to close the nutrient cycle.

5.3.3 Other food industrial WW tests

Beyond the main target of the dairy WW acidic whey stream processing, the corn milling, brewery and soft drinks, olive milling and winery WW streams are also small lab scale tested to evaluate if the 3R TH fungus biotech processing method is applicable for these WW streams with different contamination profiles and varying concentrations. The conclusions:

- a) Corn milling WW: application of the designed method for corn steep liquor liquid by-products is a possibility for nutrient recovery, but the large amount of bacterial contaminated WW can unlikely be treated economically at TRL5 scale and beyond with the designed biotech method. Furthermore, the industry already solved the full recovery, closed loop recycling and onsite reuse of process WWs by separating liquids from solids, cleaning and feed back to the main process. The industry is already using process water cleaning and exclusive blends of custom-designed WW treatment formulations for closed loops recycling and process reuse of WW, therefore it is expected that no added value can be offered by WalNUT for this case.
- b) Brewery/soft drinks WW: no success to treat sanitation WW with the designed method while nutrient recovery is economically not viable. The clean, high-quality water is the essential ingredient for all products of the beverage industry, incl. such as used for facility-level beverage production, cooling, cleaning heating waters and sanitation, but excl. use of return water and agriculture. The water use ratio is a calculated ratio of the total water usage to total beverage production at each facility. The sanitation cleaning WW stream is a small fraction of cost that closed loop recovery and reuse is already solved locally at each factory and not suitable for nutrient recovery new business model.
- c) Olive milling WW: not successful, the very high concentrations of phenol organic compounds exclude this WW from the designed method *Trichoderma* fungus and ABC adsorbent specific treatment alternative.
- d) Winery WW: no success to treat sanitation WW with the designed method while the nutrient recovery is economically not viable. The production volume of a winery is a primary factor in determining its water use ratio. Cooling water use is one of the main drivers for the range of water use ratios for distilleries, but at modern facilities already recirculating where closed loop cooling system applied. The sanitation cleaning WW stream is a small fraction of cost that is already solved locally at each winery where economy of scale production is ongoing and not suitable for nutrient recovery new business model.



5.3.4 Summary conclusion

Dairy acidic whey is a challenging WW material stream whose economical upcycling to BBF require specific solution, which is specifically applied for the case.

5.4 Comparison

5.4.1 From a technical point of view

	Alternative A 3R biotech/integrated thermal processing	Alternative B Traditional processing
Material composition	92% calcium phosphate bioapatite mineral 8% carbon	91% carbon 9% ash
Spec. surface area	80 - 120 m ² /g BET*	500 - 1200 m ² /g BET
Iodine	100 – 300 mg/kg	800 – 1200 mg/kg
Ash	1% max.	5% - 9 %
Hardness	98%	98%
Porosity	40-63,000 nm macroporous 100%	2-20 nm mesoporous 10% and microporous 90%
Pore volume	0.55 cm ³ /g	0.49 cm ³ /g
Bulk density	650 kg/m ³	500 kg/m ³
Mesh size	4x8, 6x12, 8x20, 8x30, 12x40	4x8, 6x12, 8x20, 8x30, 12x40
pH	8 – 10.5	7.5-11.5
PTEs mg/kg	Cd <0.3; CrVI nd; Hg 0.03; Ni 2; Pb 1; As <1; Cu 13; Zn 89.	Cd 1; CrVI nd; Hg 0.5; Ni 10; Pb 50; As 20; Cu 50; Zn 10.
PAHs	1 mg/kg (PAH 19 as of MS regulation)	4-6 mg/kg (PAH 16 as of EU regulation)
Phosphorus	155,000 mg/kg (35.5% P ₂ O ₅)	1,860 mg/kg
Calcium	259,000 mg/kg (36.2% CaO)	48,600 mg/kg
Application areas	High efficiently remove macromolecular organic pollutants such as from food industrial WW. Highly suitable as bio-carrier for soil biotech formulations, soil applications and low pH substance processing. Having unique macroporous structure and surface characteristics. Containing economically high phosphorus density.	Remove nano and micro chemical pollutants, water purification, gas adsorber, pharmaceutical and chemical industries, solvent adsorption. Not suitable for efficient and economical soil applications, and cannot be properly bio-formulated.
Processing temperature	850 °C material core	1000 °C material core and activation
Energy consumption	Energy self-sustaining and surplus energy producer	Highly energy demanding and intensive.
Reagent consumption	No reagents or chemicals used. Not activated, natural char.	High toxic chemicals used for activation, chemical activation with potassium hydroxide (or NaOH or H ₃ PO ₄ or ZnCl ₂)
Space requirements	Medium scale industry space	Medium scale industry space
Distance (from the source)//Transport impact	Regional short supply chain for the European origin input feed material	Transcontinental long supply chain for the tropical origin input feed material
Carbon footprint	Zero emission processing and energy independent	Highly negative environmental / climatic impacts
Adsorbent regeneration	Yes, 3-4 times, thereafter reused as soil biofertiliser.	Yes, 3-4 times, thereafter incinerated and energy converted.
Market price	€ 1500 / tons	€ 3500 / tons
* Brunauer–Emmett–Teller		



5.4.2 From a LCSA point of view

	Alternative A 3R biotech/integrated thermal processing		Alternative B Traditional processing	
Energy consumption	In economical full industrial scale the 3R integrated process is energy self-sustaining performance targeted.	-	The nano-filtration processing is highly energy intensive.	+
Reagent consumption	No reagents used. Additional food industrial by-products will also be added to the liquid medium.	-	Reagents applied.	+
Space requirements	Small industrial installation.	-	Large space requirement for anaerobic digester to be operated in economical scale.	+
Distance (from the source)//Transport impact	Regional dairy industrial solution with up to 50 km from the source.	=	The anaerobic digester is a regional dairy industrial solution with up to 50 km from the source.	=
Carbon footprint	In economical full industrial scale the 3R integrated process is zero emission performance where all material streams upcycled. High added value transformation of unexploited biomass into new products provided, perceived to be of greater quality and environmental/climate value with second life and new function that finished product becomes more practical and valuable than what it previously was.	-	Carbon footprint is not calculated, but in preliminary estimation several climate impacts to be considered at different alternative applications.	+
Waste generation	No waste generated, all material streams are upcycled and reused, nutrient and water cycles fully closed.	-	At different alternative applications different waste streams generated.	+

5.5 Brief description of the WP3 pilot no. 3, based on the previous results

Based on the previous results, 3R is developing, designing and testing a specific WP3 pilot liquid and solid state fermentation technology where the dairy food industrial WW acidic whey will be used as liquid medium during the fermentation process. Additional food industrial by-products will also be added to the liquid medium. The liquid fermenter will be inoculated by a selected agriculturally beneficial microbial strain (*Trichoderma spp*) that is selected specifically for the acidic whey biotech processing, consisting high lactose content and low pH4. The BIO-NPK-C compound BBF is humus co-composted where the technological irrigation clean water is also reused for moisture control. In this context a community of organisms fostered that by use decompose organic material and break down matter, improve soil structure, and create a prolific soil ecosystem and fertile environment for healthy plant growth. The lawful final product quality and safety will be checked. The 3R WP3 TRL5 pilot validation scales:

1. 150 L/batch liquid fermenter.
2. 500 L/batch solid fermenter.
3. Liquid separator to separate the fermented WW liquid/microbiologically concentrated solid parts.
4. Adsorber to treat the fermented liquid parts or directly the acidic whey. ABC adsorbent will be used for final treatment of the separated liquid effluent from the liquid fermentation process. The end-product is a clean irrigation water stream for horticultural application or other industrial uses.

The unique macroporous adsorbent applied (ABC Animal Bone Char or BioPhosphate) is a high P (35% P₂O₅) and Ca (37% CaO) density material, which is REACH tested and validated as of T4.5, that is the regulatory precondition of WP3 TRL5 and WP4 demo operations at <10 t/y scale.

In this context, the core problem of the dairy sector is with the 90% WW stream acidic whey, which is industrially unsolved and therefore consisting WalNUT focus and objective driven strategy, such as the



“nutrient recovery from waste water”, “re-design the value and supply chains of nutrients” and “to showcase the full potential of waste water as a raw material for biofertilisers production”. Acidic whey WW is the overwhelming largest liquid by-product stream of the dairy food industry with significant economic importance. This problematic liquid stream is converted into a biofertiliser with high nutrient recovery efficiency while environmental impacts minimised. In this context the 3R provides a biotech solution for the nutrient recovery from the main waste water WW stream acidic whey produced by the dairy food industry that is a recovered resource raw material. The total dairy WW acidic whey material treatment volume is 120 m³.

5.5.3 WP6 regulation and related key legal issues defined that affect pilot no 3

1. Dairy industrial products with EU origin only from approved EU food establishments with specific and strict hygiene requirements for food of animal origin are obligations for food business operators production, processing, distribution and placing on the market. In the specific 3R dairy food industrial processing case the acidic liquid whey by-product targeted, that is overwhelming single largest and economically most important EU dimensional stream, needs to be upcycled to convert problem into benefit.
2. Food business operators must initiate procedures to ensure that raw milk is not placed on the market if it contains antibiotic residues in a quantity that, in respect of any one of the substances referred in the regulation exceeds the authorised levels under that Regulation; or the combined total of residues of antibiotic substances exceeds any maximum permitted value. When raw milk fails to comply which is daily inspected, that raw milk must be discarded to avoid entering the dairy food processing chain, and the food business operator must promptly inform the competent authority and take measures to correct the situation. It is also important to highlight that efficient dairy industrial processing, especially cottage cheese and Greek yoghurt production, is not possible when milk containing above food limit antibiotics and pharmaceutical residuals or PTEs, that limits in the practice are not detectable.
3. Procedures based on the hazard analysis and critical control points (HACCP) principles, good hygiene rules/criteria/practices through the food chain ("from farm to fork"). (EC) No 853/2004, (EC) No 178/2002 and amendments/other EU regulations/related documents applied since 2006. New EU regulations are also underway to be implemented that are considered during tracking in WP3, incl. COM(2022) 541 final.

5.6 Conclusions

Based on the previous results, 3R is developing, designing and testing a TRL5 WP3 pilot liquid and solid-state fermentation technology where the food industrial waste water WW acidic whey will be used as liquid medium during the fermentation process. Acidic whey WW is the overwhelming largest liquid by-product stream of the dairy food industry with significant economic importance.

This problematic liquid stream is converted into useful biofertiliser with high nutrient recovery efficiency while environmental impacts minimised. The liquid fermenter will be inoculated by selected agriculturally beneficial microbial strains (*Trichoderma spp*) that are selected specifically for the acidic whey biotech processing, consisting high lactose content and low pH4. Additional food industrial by-products will also be added to the liquid medium.

A unique macroporous adsorbent is applied (ABC Animal Bone Char or BioPhosphate), that is a high P and Ca density material, and which is REACH tested as of T4.5., the regulatory precondition of WP3 TRL5 and WP4 demo operations at <10 t/y scale.

The problematic liquid stream is converted into P/Ca based multi-functional compound biofertiliser with combined effects by multi-stage processing for wide range of product functional applications (as of safety, quality and labelling compliance defined by the (EU) 2019/1009 PFC categories) and high nutrient recovery efficiency while environmental impacts minimised. The high nutrient density compound BBF BIO-NPK-C output products will be delivered to regional users, who will use it for their onsite CMC3



green compost material enrichment and improvement where the technological irrigation water is also reused.

The circular economy based 3R provides a biotech upcycling solution aiming the nutrient recovery from the unexploited main waste water stream dairy food industrial acidic whey, whose problematic WW management is transformed into resource and converted into market competitive multifunctional biofertiliser production for sustainable reuse to close the nutrient loop.



6. Nanofiltration and selective crystallisation (Precipitation, Evaporation and Flotation)

6.1 General description

Brine is a hypersaline by-product considered waste with high concentration of minerals and metals. Current practice in countries using large-scale desalination plants is to reject brine back to the sea, leading to the degradation of local fauna and flora. Extraction of materials (like Magnesium, Calcium, and Potassium salts) and high purity water recovery would contribute to the minimisation of environmental footprint of integrated desalination plants (J. Le Dirach Simon Nisan, C. Poletiko, 2005). Given its limited geological sources, Mg^{2+} has been characterised as a Critical Raw Material by the EU COM(2017)490. The pilot scale operation of the proposed seawater brine treatment is described in (Figure 6-1). The pilot is designed to treat 500 L/d of the seawater desalination brine in a semi-automatic mode and will be controlled by an advanced programmable logic controller (PLC).

6.2.1 Feedstock

Complementary to D2.1, Table 6-1 shows the concentration of major anions and cations contained in three seawater reverse osmosis (RO) desalination brine samples and in seawater.

Table 6-1. Ions concentration in three seawater RO desalination brine samples and in seawater.

Ions	Concentration in brine (mg/L) 1st sample	Concentration in brine (mg/L) 2nd sample	Concentration in brine (mg/L) 3rd sample	Concentration in seawater (mg/L) reference
K ⁺	813	874	906	480
Na	19674	17781	18230	8026
Ca ²⁺	776	740	760	450
Mg ²⁺	2736	2225	2200	1314
Cl ⁻	35400	44000	43800	27000
SO ₄ ²⁻	3820	5700	4730	4400
HCO ₃ ⁻	238	107	221	140
PO ₄ ³⁻	0.08	0.42	0.23	-
NO ₃ ⁻	0.01	1.1	0.8	-
TDS	6.85 g/100ml	7.43 g/100ml	7.48 g/ 100ml	3.45 g/ 100ml
Electrical conductivity	64.2 mS/cm ²	74.1 mS/cm ²	73.2 mS/cm ²	50.0 mS/cm
pH	7.77	7.7	7.8	7.5-8.4



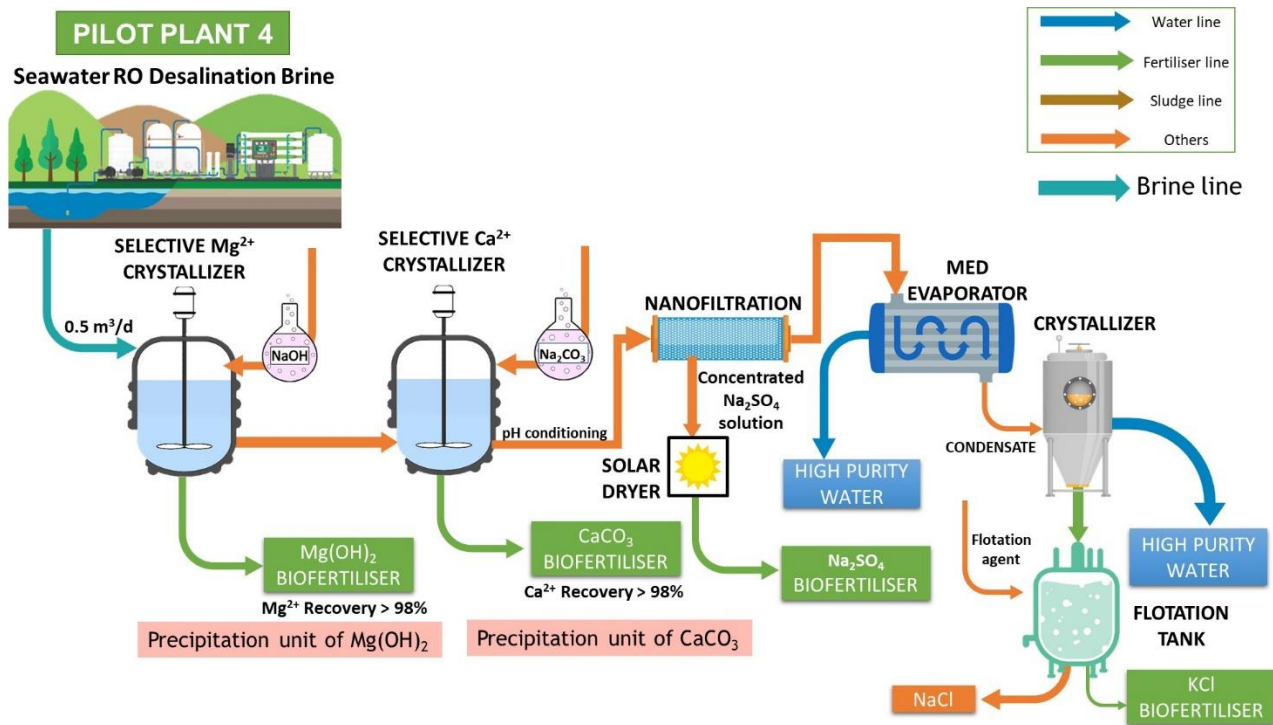


Figure 6-1. Process flow diagram of the proposed seawater brine treatment.

6.1.1 Stage 1-2: Precipitation units

In these two stages, magnesium is removed from the brine in the form of $\text{Mg}(\text{OH})_2$ and calcium in the form of CaCO_3 . Both $\text{Mg}(\text{OH})_2$ and CaCO_3 are micronutrients that can be utilised in the enrichment of bio-based fertilising products. $\text{Mg}(\text{OH})_2$ is an inorganic fertiliser and CaCO_3 is a liming material.

The brine will be transferred from the Raw Brine storage tank into the Precipitation unit of $\text{Mg}(\text{OH})_2$, which consists of a cylindrical tank equipped with an agitator, a dosing pump for NaOH (2M) addition, pH, temperature, and level sensors. NaOH (2M) will be dosed causing $\text{Mg}(\text{OH})_2$ to precipitate. The time for the precipitation of $\text{Mg}(\text{OH})_2$ will be examined between 0-5 h. After the removal of $\text{Mg}(\text{OH})_2$ slurry the supernatant liquid will be transferred to the precipitation unit of CaCO_3 , which is also equipped with an agitator, an inlet for $\text{Na}_2\text{CO}_3(\text{s})$ addition, pH, temperature, and level sensors. $\text{Na}_2\text{CO}_3(\text{s})$ will be dosed causing CaCO_3 to precipitate. The time for the precipitation of CaCO_3 will be examined between 0-3 h. After the removal of CaCO_3 slurry from the precipitation tank, the supernatant liquid will be firstly neutralised with HCl and transferred to the buffer tank of the nanofiltration (NF) Unit. (Before the NF unit the supernatant liquid will pass through bag filters and pre-filters to avoid NF fouling).

6.1.2 Stage 3: Nanofiltration unit

The NF unit aims to separate the divalent ions mainly in this case SO_4^{2-} from the monovalent ions (Na^+ , Cl^+ , K^-) of the inlet stream.

The NF system consists of a feed tank, a cartridge filter, a high-pressure pump and membranes. The feed pump circulates the brine solution from the inflow tank to the cartridge filter (pre-treatment stage). That filter has a standard $5\text{ }\mu\text{m}$ pore size and protects the main membrane. Then, a high-pressure pump passes the brine into the NF membrane, where the separation of the ions is performed. In this pilot system the operator will be able to adjust the retentate and permeate flows and at the same time recirculate the retentate into the membrane for optimal operation. The membrane used in the NF unit will be a FILMTEC NF270-4040 membrane which is a Polypiperazine Thin-Film Composite membrane.



6.1.3 Multi-effect distillation (MED)

Multi Effect Distillation (MED) is a thermal-based technology, widely used in the desalination industry, capable of treating high salinity feeds resulting in production of high purity water and brine condensate.

The MED consists of 2 effects with heat exchangers. Plate heat exchangers are installed in the system, for pre-heating the feed brine and for condensing the produced vapour. An electrical boiler supplies steam to the first effect. The system operates below atmospheric pressure, and the brine is sprayed to the first effect on top of the bundle. Hot water from boiler is running through the heat exchangers of the first effect transferring heat to the feed brine, resulting in water evaporation and brine concentration. Vapour generated by the first effect is transferred to the heat exchangers of the second effect. Thus, the necessary heat for brine vaporisation in the second effect is provided by internal heat gain, resulting in energy recovery. The concentrated brine from the first effect is directed towards the second effect for further concentration. In addition, the concentrated brine stream from the second effect is recirculated back to the first effect until the target concentration is achieved, creating this way a close loop. The vapour stream produced by the second effect is used for preheating the inlet brine passing through a heat exchanger and then it is condensed in a plate heat exchanger.

The control of the system is accomplished via a PC-based supervisory control and data acquisition (SCADA) interface. Multiple temperature, pressure and flow transmitters are connected in many positions within the system, receiving all the important data for process control. The overall control and monitoring can be achieved using SCADA. All data is collected and controlled using a programmable logic controller (PLC). The aim of the automation system is to display and record information related to the processes, the operation of the equipment etc.

The Process Flow Diagram (PFD) of the MED evaporator unit is shown in Figure 6-2 and also a 3D scheme is presented in Figure 6-3.

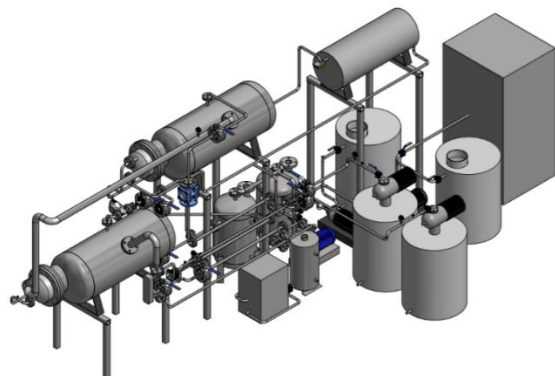
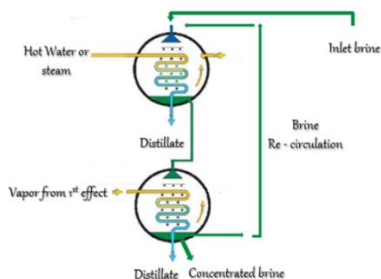


Figure 6-2. Process Flow Diagram of evaporator.

Figure 6-3. 3D scheme of the MED evaporator.

6.1.4 Crystalliser technology

A crystalliser unit will be used for further condensation of the brine and water recovery. The crystalliser is a concentrator which distillates liquids at low temperature through the combined effect of vacuum technology and heat pumping. Through a circuit, the heat pump carries out the expansion and compression of the Freon gas and yields both the necessary calories for the evaporation of the liquid and the necessary frigories for its condensation. A thermal exchange takes place in the lower part of the boiling chamber and vapors are condensed in the condenser. The distillate is drawn from the receiver and pumped through the ejector to create vacuum. The pressure variation produced is sufficient to extract both the concentrate and the distillate. The energy consumption has been measured by the electrical panel of the system. The crystalliser that will be used in this pilot can treat 0.2 m³/day. Photos of the equipment are presented in Figure 6-4.





Figure 6-4. Crystalliser unit.

6.1.5 Solar dryer unit

For the drying of all salts produced, solar dryer units (Figure 6-5) will be constructed.



Figure 6-5. Solar dryer unit.

6.1.3.4 Flotation unit

The mixed (NaCl-KCl) salt is led to the flotation cell for the separation of KCl from NaCl. For this purpose, a saturated solution of KCl and NaCl is used with Sodium Dodecyl Sulfate 1% or Hexa-decyl-trimethyl-ammonium bromide as floating agents. An incorporated agitator is mixing the solution by providing air during the whole process to create froth. Due to the hydrophobic nature of KCl, its crystals are adhered to bubbles, raised up to the surface and collected.

6.2 Alternatives

As part of the design of the previously explained brine treatment train (Figure 6-1) different alternatives regarding the process parameters (see D2.6), were evaluated to define the pilot scale operation which is going



to take place in WP3. These aspects were the type of reagent (NaOH and $\text{Ca}(\text{OH})_2$), the reagents quantity, the reaction (mixing) time, the reagents' addition method and the flotation agent.

6.2.2 Effect of pouring vs. Spraying NaOH on sedimentation speed

Different ways of reagent addition were tested in order to achieve a faster precipitation of produced $\text{Mg}(\text{OH})_2$. Besides pouring NaOH into brine, an alternative way of addition is the spray method. In this case, the precipitation of produced mineral is faster (Figure 6-6). Figure 6-7 shows two volumetric tubes with the same quantity of brine and NaOH that were left static in order to separate the liquid from the solid. It is observed that after 2.5 hours in the left tube, where NaOH is added through spray method the separation of solids from liquids is faster resulting in a precipitation with higher density (Table 6-2).

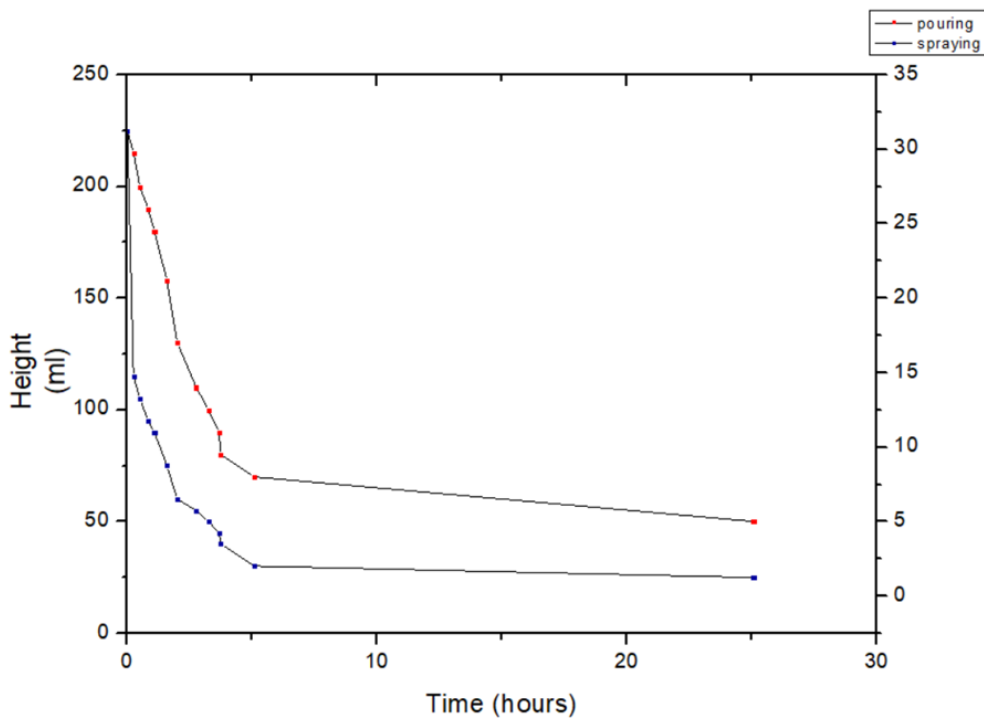


Figure 6-6. $\text{Mg}(\text{OH})_2$ slurry separation (24h) through spraying and pouring NaOH (2M).

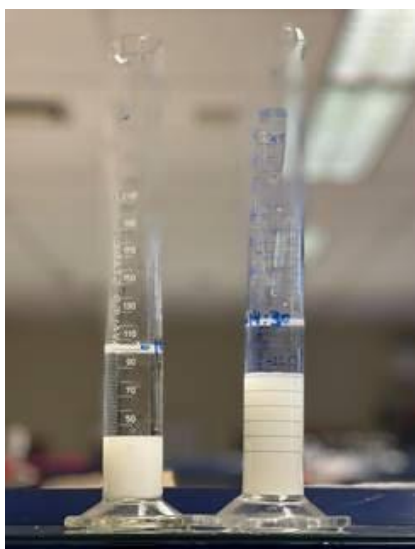


Figure 6-7. Addition of NaOH by spraying (left) - without spraying (right).

Table 6-2. Precipitate density of $\text{Mg}(\text{OH})_2$ slurry.

	Precipitate density of $\text{Mg}(\text{OH})_2$ slurry
Pouring method	~50 g/l
Spraying method	~30 g/l

Comparison

6.2.3 Comparison of NaOH vs. $\text{Ca}(\text{OH})_2$

Concerning the comparison of NaOH vs $\text{Ca}(\text{OH})_2$ addition in recovered salts purity as seen from Table 6-3, using NaOH as a reagent instead of $\text{Ca}(\text{OH})_2$ results in higher purity of recovered $\text{Mg}(\text{OH})_2$ salts.

Table 6-3: Percentage purity of solids in $\text{Mg}(\text{OH})_2$ using NaOH or $\text{Ca}(\text{OH})_2$ as reagents.

Stoichiometric quantity/Mixing Time	NaOH as reagent		$\text{Ca}(\text{OH})_2$ as reagent	
	% $\text{Mg}(\text{OH})_2$ (s) recovery	% $\text{Mg}(\text{OH})_2$ (s) purity	% $\text{Mg}(\text{OH})_2$ (s) recovery	% $\text{Mg}(\text{OH})_2$ (s) purity
90% 15'	86	88	87	86
90% 45'	89	92	87	76
100% 30'	95	89	95	71
110% 15'	99	85	99	85
110% 45'	99	93	99	67

6.2.4 Comparison of Sodium dodecyl Sulfate and Hexadecyl Trimethyl Ammonium Bromide as flotation agents on X-Ray Diffraction purity control of recovered KCl crystals

The representative diffractograms are presented in Figure 6-8 and Figure 6-9 where X-Ray Diffraction (XRD) analysis of the initial mixed salt is compared to each recovered KCl.



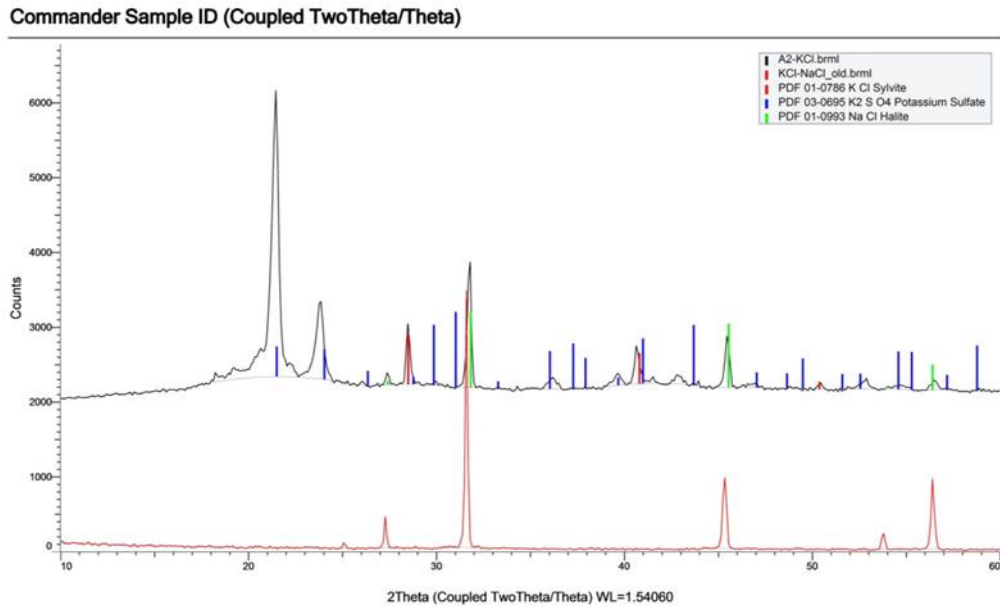


Figure 6-8: XRD analysis of recovered KCl using sodium dodecyl sulfate as flotation agent

When Sodium dodecyl sulfate is used as flotation agent there is presence of potassium sulfate which is raised up to the surface and inevitably collected (Figure 6-8).

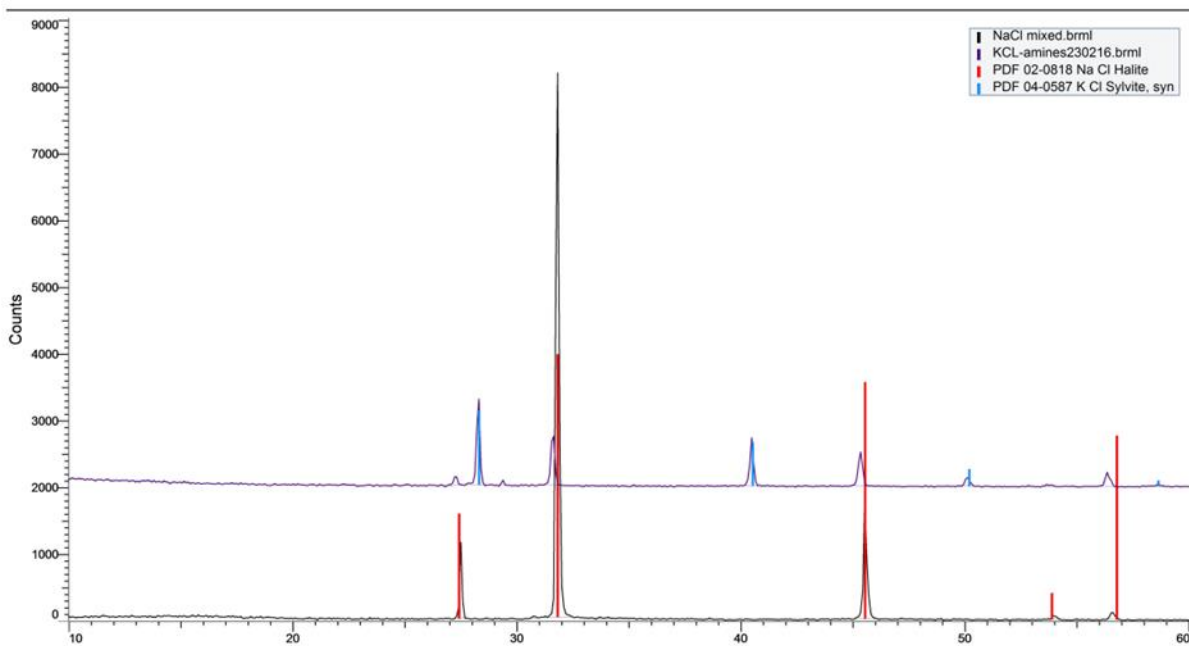


Figure 6-9: XRD analysis of recovered KCl using Hexadecyl Trimethyl Ammonium Bromide as flotation agent

It seems that Hexadecyl Trimethyl Ammonium Bromide shows high selectivity of KCl removal (Figure 6-9).

6.2.3 Environmental comparison

Regarding the addition method of NaOH the reagent's consumption is the same for both pouring and spraying method. The same applies for the energy consumption and the cost of these methods. So far, no data are available for the transport details needed in table Table 6-4.

Even though NaOH consumption is higher compared to $\text{Ca}(\text{OH})_2$ consumption for the recovery of $\text{Mg}(\text{OH})_2$, the purity of salts obtained from the reaction of brine with NaOH is higher. No differentiation appears regarding the energy consumption of the two different reagents. The cost of NaOH is lower compared to that of $\text{Ca}(\text{OH})_2$ making the process more feasible.

Table 6-4. Comparison of environmental aspects of different reagents and their addition methods .

	Addition method of NaOH		Reagents for precipitation of $\text{Mg}(\text{OH})_2$	
	Pouring	Spraying	NaOH	$\text{Ca}(\text{OH})_2$
Reagents consumption	=	=	++	-
Energy consumption	=	=	=	=
Costs	=	=	-	+
Transport	NA	NA	NA	NA

6.3 Next steps

- In this deliverable, the design of the Pilot plant 4, derives from the results of the lab-scale (200 mL, 50 L brine) experiments described in D2.6. These results contributed to the selection of the operational units that need to be incorporated in brine treatment. The preliminary design will follow the set-up included in Figure 6-1, with an important scale-up, treating 500 L of brine/day. The different scenarios that were evaluated in the previous sections are the predecessors of the design of Pilot plant 4.
- Based on the design parameters addressed in the previous sections of D2.8 the pilot components and spare parts for precipitation and flotation stages have been ordered.
- The MED and crystalliser units are already operational and occupied in a sister project and will be transferred in Lavrion Technological and Cultural Park, after necessary refurbishment, to be fully containerised with the precipitation, nanofiltration and flotation units. The refurbishment of both the MED evaporator and crystalliser are to be completed.
- Pilot plant 4 is to be constructed, following the technical results presented in this report. The Pilot plant 4 will be installed in Lavrion Technological and Cultural Park and it is expected to be ready for start-up and conditioning in a few months.
- The recovered products (Bio-Based Fertilisers (BBFs) and high purity water) will be assessed in terms of quality, quantity, and crop yield.

Some challenges that may arise before/during/after the start-up of Pilot Plant 4 could be:

- Shortage and delay in material/equipment supplies
- Time consuming NTUA administrative bureaucracy.

6.4 Conclusions

NTUA has completed the design of Pilot Plant 4 for brine treatment (capacity 500 L/d), using inputs from completed lab-scale tests. Pilot Plant 4 will be tested in the Technological Park of Lavrion, Attika (Greece). The Pilot components and spare parts have been ordered for precipitation and flotation stages and existing NF, MED and crystalliser units are to be refurbished. Technologies proposed are based on recovery of



economically prime important nutrient resource-based products and Zero Liquid Discharge principles where two different outputs are generated: value-added solids (salts and other chemicals) and high purity reclaimed water. Experimental results show that the reagents' quantity is the most important parameter that affects the minerals' recovery while stirring time is not such a determinant factor. Recoveries achieved are more than 90% and in some cases reached 100%. When NaOH is used for $\text{Mg}(\text{OH})_2$ precipitation, the highest purities (~93% and 92%) are achieved after longer stirring time, independently of the amount of the reagent added. On the contrary, when $\text{Ca}(\text{OH})_2$ is used, recovered salt purities are higher (~86% and 85%) when the mixing time is shorter. As for the recovered CaCO_3 it seems that its purity (~99%) is not affected by the examined experimental conditions. KCl recoveries and purity vary depending on the amount and the type of flotation agent used.

The expected recovery of salts and high purity water from the treatment with the proposed recovery train of 500 L seawater desalination brine per day is:

- $\text{Mg}(\text{OH})_2$ (5.0 kg/m³)
- CaCO_3 (1.0 kg/ m³)
- KCl (1.6 kg/ m³)
- Na_2SO_4 (6.0/kg/ m³)
- NaCl (20.0 kg/ m³)
- water (0.8 m³/m³).

After the operation of Pilot Plant 4 and the Life cycle analysis in the framework of WalNUT-WP7, the technical and environmental feasibility of the proposed innovative approach will be verified together with the minimisation of the water treatment costs.



7. Ion exchange and membrane contactor process

7.1 General description

Nutrient recovery from waste water, mainly nitrogen and phosphorus, is considered one of the most important ways to increase the nutrient circularity, extracting and concentrating the nutrients present in this source to obtain a marketable product such as ammonium salts, struvite or hydroxyapatite. Regarding nitrogen recovery, different technologies have been developed and implemented in the last decade such as stripping, microbiological processes, ion exchange or membrane technologies which have different TRL and final products. Although all of these processes could fit with the aim of the WalNUT project, the technology implemented by CETAQUA is based on membranes (Figure 7-1).

Hollow fibre membrane contactor (HFMC) is one of the most promising technologies for nitrogen recovery. It is a gas-permeable membrane, normally made of polypropylene, which offers a high contact surface between two streams. Due to its hydrophobicity only gas substances are able to cross the membrane, so, in case of ammonia, the ammonium rich solution should have a high pH (more than 8.6) to transform ammonium to free ammonia as gas. Once free ammonia crosses the membrane, it is captured by an acid solution, usually sulphuric, phosphoric or nitric acid, which will determine the marketable salt obtained: ammonia sulphate, ammonia phosphate or ammonia nitrate. Figure 7-1 shows a scheme of the technology

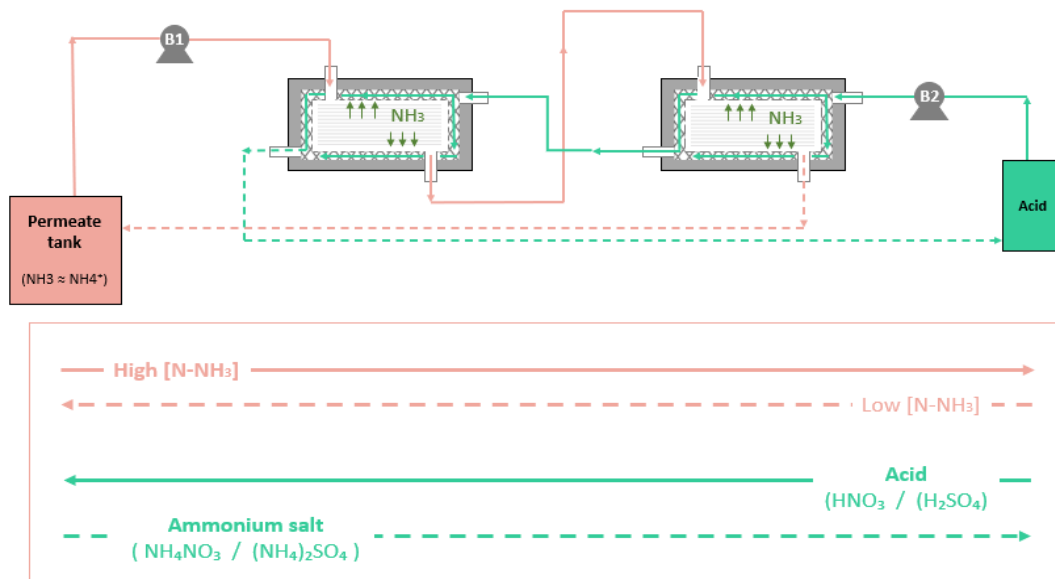


Figure 7-1. HFMC N recovery system proposed by Cetaqua.

The main advantages of this technology are:

- Low energy consumption due to the driving force is the ammonia concentration in both sides of the membrane.
- Low space requirements.
- Modularity.
- High ammonia selectivity because its hydrophobicity only permits the pass of gas substances.
- High quality products due to membrane hydrophobicity avoid the pass of heavy metals or PTFEs or OMPs to the final product.
- High range of ammonia concentration in the feedstock.



Although the ammonia content of the feed solution has no influence on the recovery efficiency of the membrane, it has an important role in economic aspects of the technology. The lower ammonia concentration in the feed, the higher the reagents costs to increase the pH per gram of ammonia recovered. Because of that, it is necessary to select the most concentrated ammonia stream of the WWTP, which is the anaerobic supernatant. The ammonia concentration of this stream normally varies from 600 to 800 ppm of N-NH_4^+ . However, a previous step of ion exchange with natural zeolites is included in the treatment train (Figure 7-2). Including this technology has two main positive impacts: (1) higher concentration leads to better performance in the HFMC; (2) Ion exchange works in adsorption-regeneration cycles. The regeneration step produces a new stream which is at higher pH which is sent to HFMC preventing pH modifications in WWTP streams as well as allowing the recycle of the regenerant solution which reduces the chemical consumption.

Zeolites are a natural aluminosilicate microporous adsorbent able to capture the ammonium of the feed solution by ion exchange. After that, a regenerant solution, mainly NaOH or NaCl, is put in contact with the zeolite to revert the initial condition favouring the release of the ammonium. However, due to the high pH of this solution, ammonium is present as free ammonia. Figure 7-2 shows a scheme of the process.

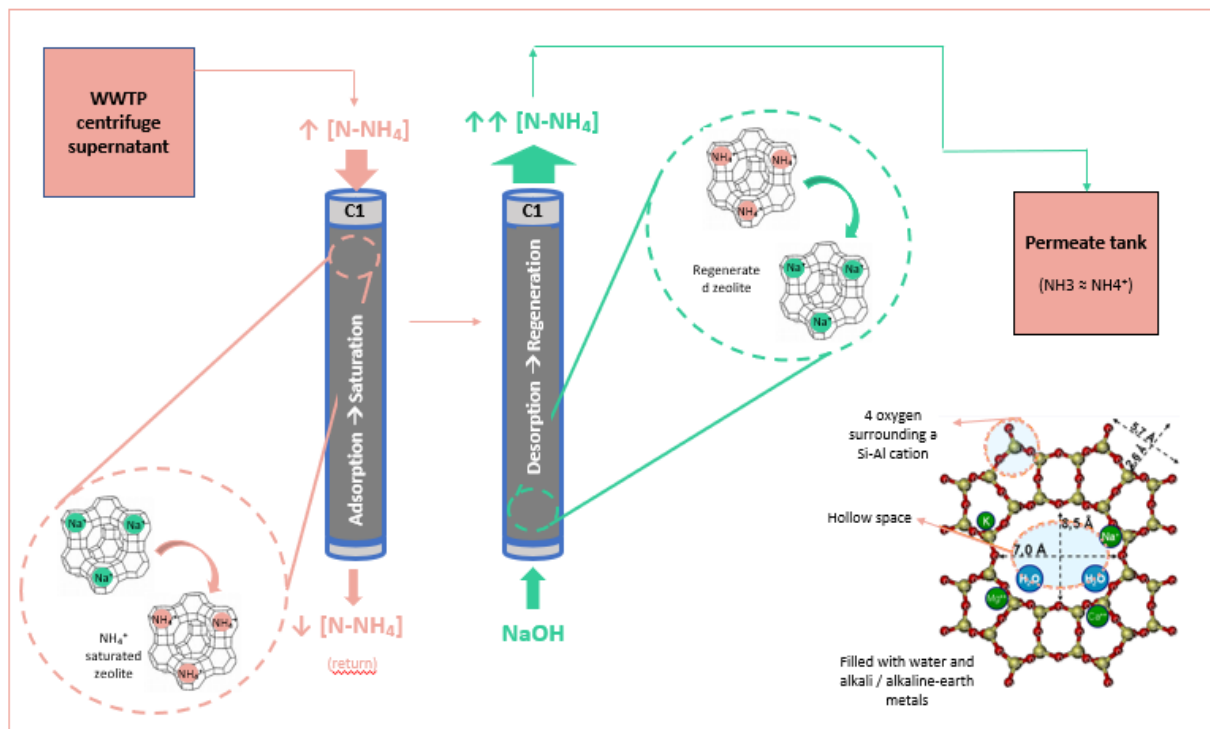


Figure 7-2. Ammonia concentration treatment proposed by Cetaqua.

Furthermore, the treatment train has another unit, the pre-treatment, which is usually needed to remove the possible solids present in the anaerobic supernatant. In this case, the design of this pre-treatment is one of the aspects evaluated in this deliverable, so the results and conclusions regarding this point are included in the following sections.

7.2 Alternatives

Involved in the study of the operation of the previously mentioned treatment train attending to different parameters such as the acid concentration, the flow rates, pH, etc. (see D2.7), different alternatives were studied to define the pilot scale operation which is going to take place in WP3. These aspects were the feedstock and the regenerant.

7.2.1 Feedstock

As the pilot plant is going to be located in Galicia, a region in the North-West of Spain, different alternatives were evaluated in order to select the best location for it. For that reason, two WWTP were evaluated (see the stars in Figure 7-3).

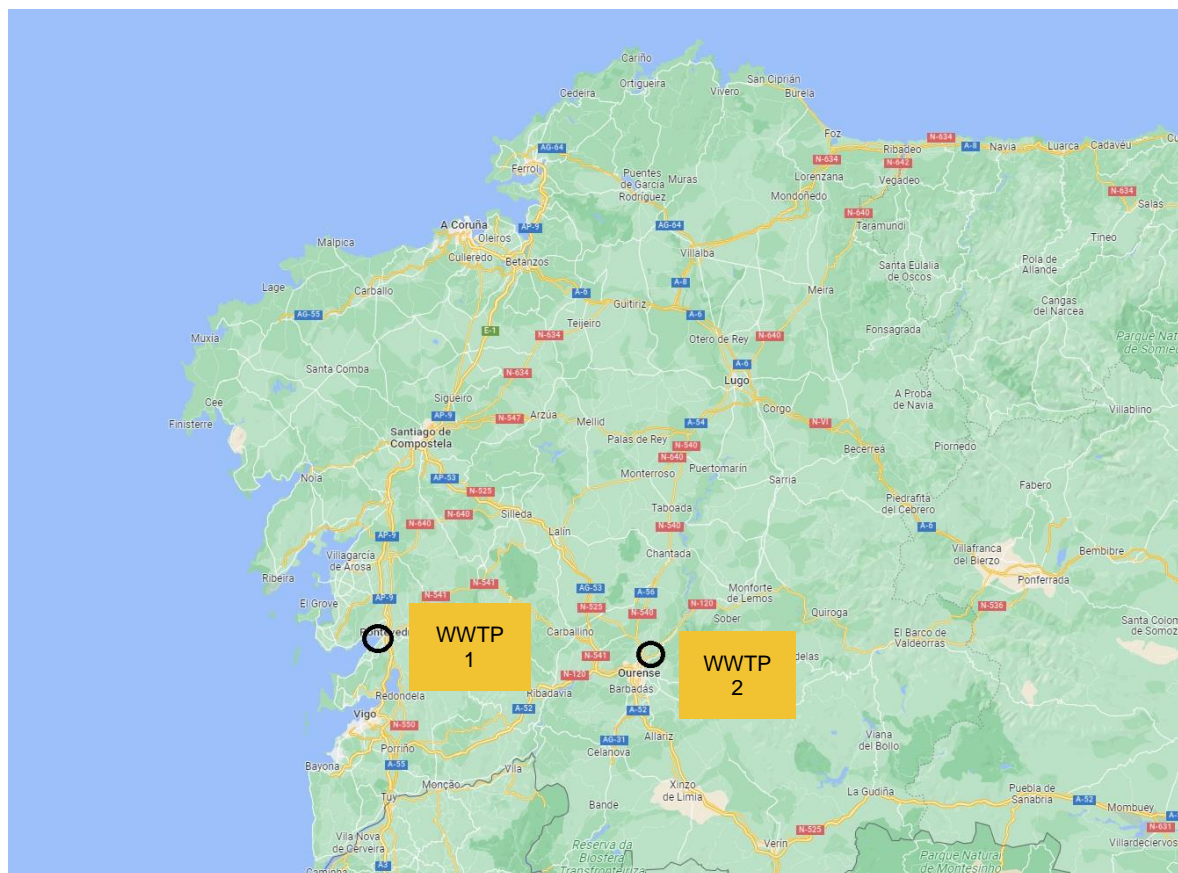


Figure 7-3. WWTP locations evaluated for pilot 5.

Although both WWTP have a similar size (around 51,000 m³/d) and present an anaerobic digestion step, their location as well as their treatment process are different. WWTP 1 is located in the coast, so the salinity has a representative impact over the process. In contrast, WWTP 2 is far from the coast, so this parameter is not important. Furthermore, the process is quite different:

- WWTP 1 presents a Moving Bed Biofilm Reactor (MBBR) for nutrient and organic matter removal alternating aerobic and anoxic conditions. The main characteristic of this treatment is the presence of plastic carriers where the biofilm is grown, increasing the contact surface bacteria-water, reducing the space and the contact time needed to reach the contaminants removal.
- WWTP 2 in contrast, a sequential process is applied. The first step is filling and aeration to remove the organic matter and to oxidise the ammonium to nitrate; a second step of settling, creating an anoxic space to favour the denitrification and, the last one of emptying.

Both anaerobic digestion supernatants were characterised in terms of COD, nutrients, alkalinity, pH and solids, being the solids and the nitrogen content.

7.2.2 Regenerant

Another aspect evaluated was the reagent used for the regeneration of the ion exchange column. As it is previously mentioned, in the regeneration process, the ammonium is released and the space of this ion should



be filled with a new one being sodium, the most common. For that reason, two different reagents were evaluated: sodium hydroxide and sodium chloride.

To evaluate this both options, using the same feedstock (from WWTP 2) and the same operational conditions (feed flow rate of 88 mL/min, 0.35M), the ammonium was adsorbed in the adsorption step and its desorption was carried out using both regenerants to compare it. The zeolites set-up is shown in Figure 7-4.



Figure 7-4. Zeolite set-up built at CETAQUA's facilities.

Although the most important aspect to evaluate the best regenerant will be the ammonia release during the desorption step, it is important to add that the price of sodium chloride is slightly lower than the sodium hydroxide so, in case of similar performance, the economic aspect will be also considered.

7.2.3 Pre-treatment

The final aspect evaluated for the scale up was the pre-treatment. The anaerobic supernatant could present suspended solids that could provoke undesirable effects over the column or the membranes such as clogging or favouring the growth of microorganisms in the column. Because of that, two alternatives were evaluated to remove most of these solids in order to protect the treatment train.

- **Settler:** the first low-cost option is to include a settling step to remove the solids present in the stream. For that purpose, a sludge sedimentation test called V30, which represents the volume of the range occupied in 30 minutes of sedimentation was carried out. After these 30 minutes, the supernatant was analysed in terms of suspended solids.
- **Filtration unit:** the second option is to add a filtration unit. It consists of two low-cost filters of 20 μm and 5 μm in series (Figure 7-5). As in the settler case, the solid content of the effluent was also analysed.





Figure 7-5. Filtration unit used for WW pre-treatment.

7.3 Comparison

7.3.1 Feedstock

The feedstock used in this research is the supernatant of the anaerobic digestion. In order to choose the best one to apply the technology both streams were characterised. The results are shown in Table 7-1.

Attending to the characteristics of each anaerobic supernatant, the best option is the one of WWTP 2 because of its lower solid content which offers new low-cost possibilities to remove them in the pre-treatment; and its higher ammonia content. Higher ammonia content increases the recovery potential and reduces the costs per kg of recovered nitrogen.

Table 7-1. Feedstock comparison of WW used in pilot 5.

	WWTP1	WWTP2
COD _t (mg O ₂ /l)	2912	3253
COD _s (mg O ₂ /l)	284	2809.5
N _T (mg N/l)	438.5	695
N _{Ts} (mg N/l)	-	468
P _T (mg P/l)	55.5	13.2
NH ₄ (mg N-NH ₄ /l)	28.05	443
PO ₄ ³⁻ (mg P-PO ₄ /l)	0	0.161
NO ₃ ⁻ (mg N-NO ₃ /l)	0.588	5.54
SO ₄ ²⁻ (mg S-SO ₄ /l)	73.05	122



pH	6.89	6.75
Alkalinity (g CaCO ₃ /l)	0.5	3.2
TS (g/l)	2.79	1.44
VS (g/l)	1.67	0.87

7.3.2 Regenerant

To evaluate the best regenerant for the Ion Exchange unit, sodium hydroxide and sodium chloride performance were evaluated. Figure 7-6 represents the ammonia retained in the column release in the desorption step using two solutions: 0.35M NaOH and 0.35 M NaCl, representing the same amount of sodium, ion which replace the ammonium released. Although the price of NaCl is lower than the NaOH ones, the consumption is higher (20.5 g NaCl instead of 14 g NaOH for a 0.35M solution) and the results show a better performance using NaOH. Despite at the beginning of the desorption step the release is slightly higher using NaCl, after a bed volume (BV) of 4, the tendency is quite different. Using NaOH as regenerant, the release increases in 3 BV from 20% to 70%, while, using NaCl, this parameter changes from 20% to 40%.

The better performance of NaOH as a regenerant determined its use during the experimentation carried out in this work package. However, a combination of both regenerants will be studied in the pilot plant task (WP3) looking for the optimum conditions in terms of ammonia release and operational costs.

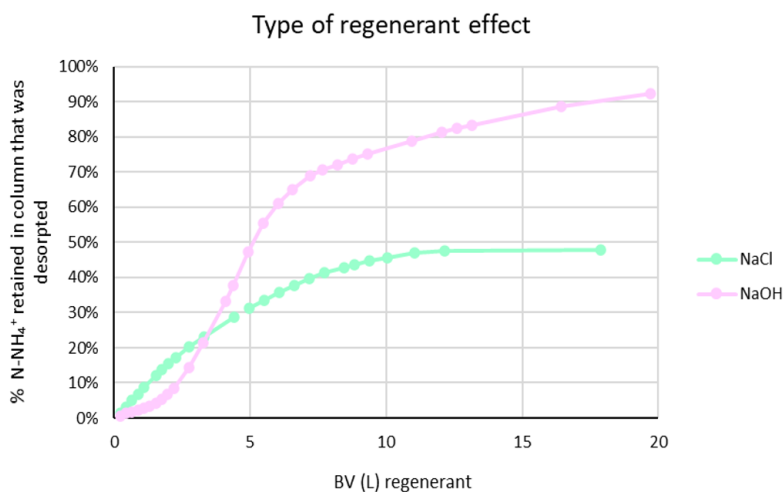


Figure 7-6. Regenerant type effect over desorption process.

7.3.3 Pre-treatment

In order to evaluate the most suitable pre-treatment, the first test carried out was related to the sedimentability of the sludge by a V30 test. A good anaerobic supernatant sedimentability will offer the possibility to use a settler as pre-treatment for solids removal. However, as it can be seen in Figure 7-7, the stream sedimentability was minimal because most of the solids are suspended.





Figure 7-7. Sedimentation test performed for pre-treatment selection.

Because of that, the alternative was to use a filtration unit. Despite most of the solids are suspended and thin, a solid reduction from 1.54 to 0.51 g/l is achieved with the filtration. The final concentration is low enough to use in the membranes, taking also into account the indirect filtration which takes place in the ion exchange unit.

7.3.4 Environmental comparison

Apart from the technical point of view, which has been evaluated in the previous sections, other environmental aspects must be analysed and taken into account into the choice of the feedstock, regenerant and pre-treatment. Table 7-2 shows the main conclusions of the environmental aspects.

Table 7-2. LCSA for ion exchange and membrane process pilot.

	Feedstock		Regenerant		Pre-treatment	
	WTP1	WWTP2	NaOH	NaCl	Settler	Filtration
Reagents consumption	N/A	N/A	-	+	++	-
Energy consumption	-	+	N/A	N/A	-	+
Costs	N/A	N/A	+	-	+	+



Transport	+	+	N/A	N/A	N/A	N/A
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Related to the environmental aspects of the feedstock selection, the main difference is due to the energy consumption. Although the process is similar in both cases, to obtain the same amount of recovered nitrogen, we would need more volume from WWTP 1 because of its lower ammonia concentration.

Regarding the regenerant, the NaCl consumption will be higher, but its cost is lower than NaOH. However, the difference between each performance during the regeneration step favours the use of NaOH due to the pH raise which later improves the performance of the membranes.

Finally, the low sedimentability of the stream will require adding more reagents to promote sedimentation, which also necessitates the use of filtration from an environmental point of view

7.4 Next steps

According to the results presented in this deliverable and in D2.7, the main parameters have been studied and selected to design the pilot-scale plant as well as start its operation, the preliminary design will follow the set-up included in Figure 7-8, with an important scale-up, treating 800 l/day.

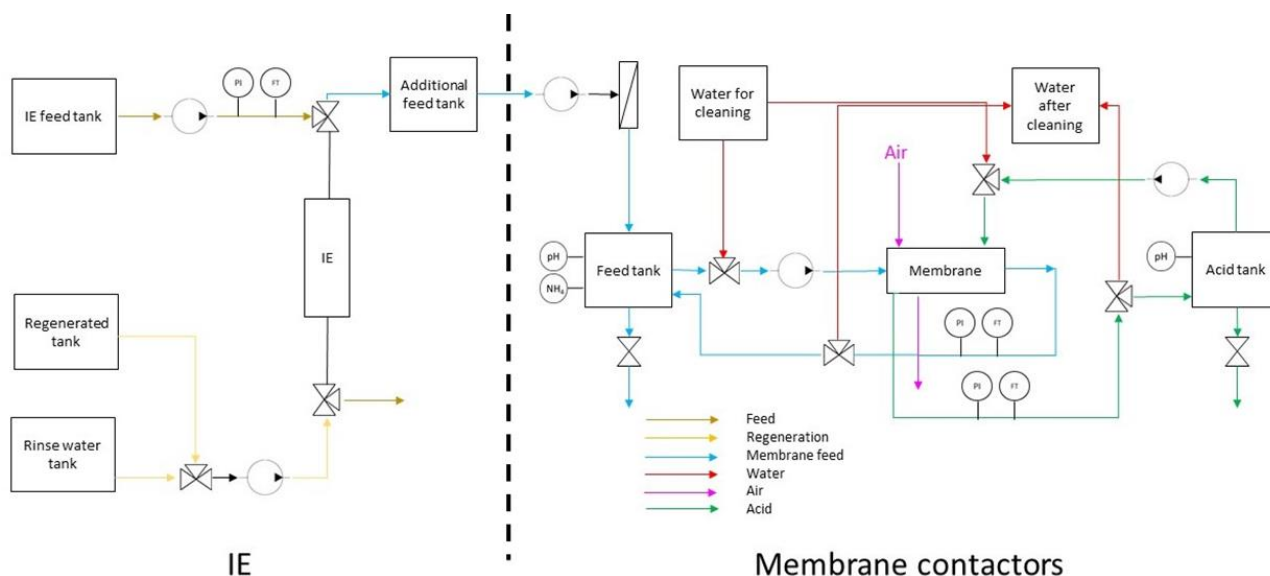


Figure 7-8. Preliminary design for scaled-up technology.

The treatment train will be like the one used in the laboratory including a pre-treatment with a filtration system to remove the solids. This pilot will be located in WWTP2 which provides higher flow rates and higher concentration of ammonia favouring the optimal recovery in terms of costs.

7.5 Conclusions

The results presented in this section offer the following conclusions:

- Pilot should be located at WWTP 2 because of the distance with our laboratory, the higher nitrogen concentration and the lower solids concentration.
- The regenerant used will be NaOH because of its better performance in terms of ammonia release during the regeneration process.
- The pre-treatment that is going to be implemented at pilot scale is the filtration unit because the lower stream sedimentability avoids the possibility to use a settler as an alternative for solids removal.



8. Conclusions

8.1 Pilot 1 conclusions

Pilot 1 will be located in WWTP3 (dairy industry) and a hybrid system of photoautotrophic and heterotrophic microalgae cultivation was the microbiological process selected for implementation at pilot scale.

8.2 Pilot 2 conclusions

Waste water collected at the Aartselaar WWTP site was chosen to carry out the pilot HRAS/CS process, due to its representativeness of the other collected streams and to its appropriate BOD/ NH_4^+ -N ratio of 3,4. It was also decided to use zeolites in their original ionic state (Z-O) and the 1-A and A-R-A N-recovery methods for the pilot-scale adsorption system.

8.3 Pilot 3 conclusions

Acidic whey and additional dairy by-products will be used as liquid medium during the fermentation process and the liquid fermenter will be inoculated *Trichoderma spp.* strains.

High nutrient density compound BBF BIO-NPK-C output products will use for onsite CMC3 green compost material enrichment.

8.4 Pilot 4 conclusions

Main advantages of the technologies proposed for pilot 4 scale-up are:

- Valuable micro and macronutrients recovery along with water recovery.
- Recovery rates of Mg, Ca are higher than >95 %.
- Recovery of high purity products ($\text{Mg}(\text{OH})_2$, CaCO_3).

8.5 Pilot 5 conclusions

Pilot 5 should be located at WWTP 2, the regenerant used will be NaOH and filtration is chosen as the pre-treatment selection.



9. References

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