

# NEREUS



Circular Economy

## Resource Recovery Final Report

D2.4.1 Recovery & optimization of resources



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# NEREUS FINAL REPORT

## D2.4.1 Recovery & optimization of resources

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# 1 Introduction

The NEREUS (New Energy and Resources from Urban Sanitation) Project is an Interreg 2 Seas project with a consortium of 8 project partners across the United Kingdom, the Netherlands, Belgium, and France, working on the goal of recovering valuable resources from domestic wastewater. The partners working on this project were VITO NV, DuCoop CVBA, water-link, Agglomeration of Saint-Omer (CAPSO), HZ University of Applied Sciences, University of Portsmouth Higher Education Corporation, Southern Water Services Ltd and Evides Industrial Water B.V.

The project was born from a combination of global pressures, such as the scarcity of freshwater and finite resources, as well as the desire to increase the reuse of wastewater. To achieve this, the project aimed for the adoption of technologies that recover important resources and enable the principal of a circular economy in the 2 Seas region<sup>1</sup>.

The focus was to treat municipal/domestic wastewater, in order to transform it into valuable resources, and/or to efficiently remove micro-pollutants, resulting in reusable water as a final product. Hence, in order to achieve the desired objectives, NEREUS ran from October 2017 to December 2021, and during that time, focused on the recovery and reuse of water, resources (e.g. nutrients), and energy.

As domestic wastewater contains finite nutrients in its composition, such as phosphorus, nitrogen, potassium, and calcium, it offers significant potential for reuse. Additionally, it also contains energy and heat, that can be used as a sustainable source of energy in order to reduce CO<sub>2</sub> emissions. Therefore, wastewater treatment can lead to the recovery of these important environmental assets, along with water reuse as irrigation, process, or even drinking water. Thus, NEREUS partners have set up several demo cases to investigate and demonstrate these possibilities.

This report presents and discusses results achieved among project pilot partners, Evides, Southern Water and CAPSO, who aimed to recover valuable resources from their wastewater streams. It also compares results regarding technologies used, recovered products, product quality and process optimization.

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<sup>1</sup> 2Seas region: Covers coastal areas of England, France, Belgium (Flanders) and the Netherlands which are connected by the Channel and the North Sea



## 2 Pilot plants

As mentioned, this report will investigate the resources recovered by the project partners Evides (NL), Southern Water (UK) and CAPSO (FR). In order to be able to recover resources from urban wastewater, each partner designed, built and operated a pilot scale wastewater treatment plant (WWTP) upon which they could perform their research. These three particular pilot plants varied greatly in size and focus; Evides has had two pilot plants over the course of the project which tested numerous technologies to recover many resources. Southern Water took a different approach with a package plant design aimed at recovering two specific products and CAPSO after some changes in their approach, focused on recovery of dried sludge from an existing full scale wastewater treatment plant.

### 2.1 Evides Industry water

Evides is a Dutch water company involved in many aspects of water treatment, such as producing drinking and process water and treating domestic and industrial wastewater. Along with this, they develop, with their partners, sustainable solutions to recover valuable resources. In the NEREUS project, it was the branch Evides Industry Water who were a project partner and who ran the pilot plant.

Evides focused on the recovery of all three central products with their treatment train. The pilot was initially located in a commercial area in Rotterdam with the aim of delivering recovered resources and irrigation water back to an urban farming restaurant (*Uit Je Eigen Stad*). However, due to changes in circumstances involving the restaurant that were outside the control of Evides, the pilot location was moved to Harnaschpolder, Den Hoorn, where resources are recovered from a municipal wastewater treatment plant. It is also important to note that the first phase of Evides' work, which consisted of cellulose recovery, was carried out at Schiphol's wastewater treatment plant.

#### 2.1.1 Stream profile

The term stream profile refers to the characteristics of the influent stream that is entering the pilot location and from which the resources will be recovered. As previously introduced, domestic wastewater was the chosen wastewater type to be treated. It can be divided into different streams according to their origin and composition: black water, which is water from toilets, and grey water, from showers, laundry, and kitchen (de Graaff et al., 2010; Luostarinen et al., 2007).

According to de Graaff *et al.* (2010), these sources should be treated according to their quantity and composition, in order to achieve a successful resource recovery. Table 1 presents general compositions of both black and grey water. In this report, for each plant, the type of wastewater is identified, and its composition is presented.

*Table 1 - Average characteristics of domestic wastewater, black water, and grey water*

<b>Parameter (mg/L)</b>	<b>Domestic wastewater</b>	<b>Black water</b>	<b>Grey water</b>
BOD	350	300 – 600	100 – 400
COD	750	900 – 1500	200 – 700
Total nitrogen (TN)	60	100 – 300	8 – 30
Total phosphorus (TP)	15	40 – 90	2 – 7

*Note.* Adapted from Henze & Yves, 2008

The stream profile of the Evides pilot plant can be classified as domestic wastewater. The characteristics of the *Uit Je Eigen Stad* and Harnaschpolder streams can be found in Table 2 and 3, respectively. A view inside Evides' pilot plant, in Den Hoorn, is presented in Figure 1.

*Table 2 - Uit Je Eigen Stad stream profile*

<b>Parameter</b>	<b>Unit</b>	<b>Value</b>
Average Daily Flow	m <sup>3</sup> /h	2.9
TSS	mg/L	25
COD	mg/L	58
BOD	mg/L	18
Total Kjeldahl nitrogen (TKN)	mg/L	15
Total nitrogen (TN)	mg/L	16
Total phosphorus (TP)	mg/L	1.4

*Table 3 - Harnaschpolder stream profile*

<b>Parameter</b>	<b>Unit</b>	<b>Value</b>
Average Daily Flow	m <sup>3</sup> /h	2.9
TSS	mg/L	270
COD	mg/L	550
BOD	mg/L	240
Total Kjeldahl nitrogen (TKN)	mg/L	55
Total nitrogen (TN)	mg/L	67
Total phosphorus (TP)	mg/L	8



Figure 1 - Evides' pilot plant in Den Hoorn

### 2.1.2 Recovered resources

The Evides Industry Water pilot plant aimed to recover all central resources with their treatment train, but, for the benefit of this specific report; the pilot retrieved the nutrients phosphorous, nitrogen and carbon (in the form of cellulose). Evides have proven in previous research projects that the named resources can be recovered with mainly physical and chemical processes, but this also resulted in a high energy and chemical consumption rate. It is hoped that if more biological processes are employed, the treatment train can be more sustainable. The target for recovery during the pilot phase was 25% recovery of phosphorous, 25% recovery of nitrogen (in the form of algae pigment) and finding a suitable solution to reuse recovered cellulose as a carbon source (C-source).

The project planning was to recover phosphorous from electro coagulation (EC) sludge in the form of  $\text{PO}_4^{3-}$  (phosphate). For nitrogen, the aim was to remove it from the wastewater stream via reverse osmosis (RO) and later use it to cultivate algae which would accumulate nitrogen and produce a valuable pigment, *phycocyanine*. A carbon source was intended to be recovered in the first train, as cellulose, via an enzymatic conversion.

## 2.2 Southern Water

Southern Water (SW) is an English private utility company that serves areas such as Sussex and Kent, and that treats and recycles 700 million liters of wastewater across 368 treatment plants every day. During the NEREUS project, Southern Water focused solely on the recovery of nutrients from their wastewater streams. The bio-solids produced during their treatment process is already recovered and used as a fertilizer by farmers. However, the pilot location in this project will focus on recovering fertilizer in a crystalline form and the commercial and environmental impacts of this.

The pilot location was chosen as the site of Peel Common, located in Fareham, due to feasibility considerations such as a sludge dewatering concentration of above 55mg  $\text{PO}_4\text{-P/L}$ , current struvite problems and enough space to house the pilot system.

### 2.2.1 Stream profile

The influent stream from which the nutrients will be recovered can be classified as municipal wastewater, more specifically as sludge liquor from a wastewater treatment plant (WWTP). In Table 4, the characteristics of the stream can be found. These characteristics were provided by *Royal Haskoning DHV*, a company that designed and operated the pilot at Southern Water’s Peel Common site. One week of sampling was performed on the influent stream, in April 2018, in order to determine the water quality.

*Table 4 - Peel Common influent stream profile*

Parameter	Unit	Average	Maximum	Minimum
pH	-	5.56	5.97	5.31
Conductivity @ 20°C	(mS/m)	287	318	266
Total phosphorus	mg/L	142	177	118
Phosphate (PO <sub>4</sub> – P)	mg/L	122	151	94
Phosphate (PO <sub>4</sub> – P)	mmol/L	3.92	4.87	3.02
P sludge (1mg PO <sub>4</sub> is 1.55mg FePO <sub>4</sub> sludge)	mg/L	95	124	114
Alkalinity, total as CaCO <sub>3</sub>	mg/L	1.154	1.415	966
Alkalinity, total as CaCO <sub>3</sub>	mmol/L	19	23	16
Total Calcium	mg/L	284	348	227
Total Calcium	mmol/L	7.1	8.7	5.7
Total Magnesium	mg/L	41	44	38
Total Iron	mg/L	22	24	20
Iron, dissolved	mg/L	17	24	0.44
Iron sludge (1mg Fe(II) is 1.95mg Fe sludge)	mg/L	9.8	38	0
Suspended Solids	mg/L	928	2,050	278

van Duyvenvoorde & Verhoek (2018, p. 18)

Figure 2 presents two photographs of Southern Water’s pilot: the reactor used, *Crystalactor*<sup>®</sup>, on the left, and recirculation line on the right.



Figure 2 - Photographs of Southern Water's pilot in situ

(Randall & Hossain, 2021)

### 2.2.2 Recovered resource

Southern Water focused on recovering phosphorus in the form of calcium phosphate  $\text{Ca}_x(\text{PO}_4)_y$  and magnesium ammonium phosphate,  $\text{Mg}(\text{NH}_4)\text{PO}_4 \cdot 6\text{H}_2\text{O}$ , also known as struvite. Following the NEREUS project objectives, the company aimed to validate a method for phosphorus recovery that did not need a large range of chemicals and contributed to a circular economy.

According to *Royal Haskoning DHV's* report, 2018, sludge liquors, that originate from sludge treatment processes at a WWTP, offer a good source for nutrient recovery while treating water, and combining operational cost reduction. Therefore, the nutrients, in both forms, were recovered from a sludge liquor by using a pellet reactor, in two different routes, and are meant to be used as sustainable fertilizers for agriculture.

## 2.3 CAPSO

The Agglomeration Community of the Pays de Saint-Omer (CAPSO) is a public inter-municipal cooperation, composed of 53 municipalities, having about 105,000 inhabitants. The community has continuously improved the quality of waste service while optimizing costs through economies of scale, all contributing towards sustainable development (CAPSO, n.d.).

CAPSO's pilot worked by installing new equipment in the Saint-Omer wastewater treatment plant. The pilot focused on the sludge stream coming from the WWTP. The goal was to improve sludge siccidity and quality for safer storage, reduced pollution potential and use as a more effective fertilizer.

This goal would reduce sludge volume, recover nutrients and help local farmers who could spread it on their fields.

### 2.3.1 Stream profile

The feed of CAPSO's treatment train was the final sludge obtained from the Saint-Omer WWTP, therefore being composed of organic and mineral material. The water content of the sludge was of 75%, which is an important influent parameter for CAPSO's case.

Figure 3 presents a view of Saint-Omer wastewater treatment plant, the site where CAPSO optimized sludge recovery.



*Figure 3 - Saint-Omer wastewater treatment plant*

(Courouble, 2021)

### 2.3.2 Recovered resource

CAPSO focused on recovering dry sludge, from a local wastewater treatment plant, in order to be used as soil conditioning at local farms. The main objective was to improve the sludge process at the WWTP, specifically the dryness of the sludge and the height of the sludge stack.

To achieve their goal of meeting regulatory requirements, CAPSO investigated the use of different limes, a dewatering product, and also the improvement/changes in the plant's installation. According to France's regulation, the dried sludge should have a siccity (dryness) level of  $\geq 30\%$ .

### 3 Methods

In order for the intended resources to be recovered from the source wastewater, a treatment train made up of various technologies was designed and installed at each pilot plant. The complexity of the treatment train was dependent on the resource or in some case number of resources to be recovered. In this chapter, a process flow diagram and technology description per pilot partner is reported.

#### 3.1 Evides Industry Water

##### 3.1.1 Process flow diagram

Figure 4 shown below contains Evides' process flow diagram involved in the nutrient recovery (highlighted in green in the diagram): PO<sub>4</sub>, nitrogen product and cellulose.

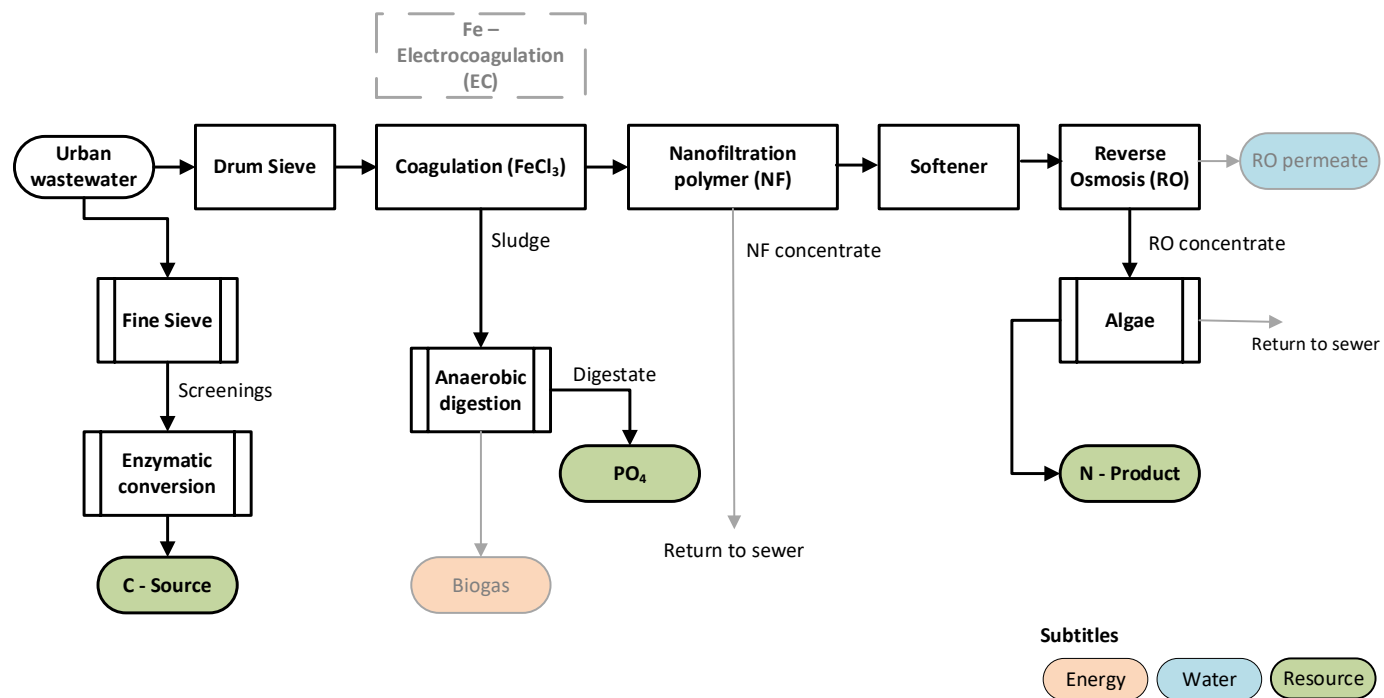


Figure 4 - Evides' process flow diagram for resource recovery

##### 3.1.2 Technologies

A different set of technologies was used for the recovery of each resource, as shown in Figure 3, and explained in chapter 1.1.2. These technologies are described below:

- **Sieves:** Sieves are perforated metal plates, commonly applied as a primary treatment, as they aim to remove large particles from the wastewater (VITO, 2010). Therefore, to fulfil the EU Council Directive criteria, 1991, it must remove at least 20% of organic matter, measured as biological oxygen demand (BOD), and 50% of the total suspended solids (TSS)(Council Directive, 1992).

In the Evides pilot plant, two different types of sieves were used: drum and fine sieve. The drum sieve consists of a rotating perforated drum, through which the fluid passes, whilst retaining the solid particles (Trevi, n.d.; VITO, 2010). This technology is used as primary treatment for both phosphorus and nitrogen recovery. The fine sieve (mesh size <0,35 mm) is used in the cellulose recovery process. When this type of sieve is used on raw sewage, cellulose fibers, that are mainly originated from toilet paper, are recovered with high purity (Ruiken et al., 2013).

- **Electro-coagulation (EC):** Consists of pairs of metal sheets called electrodes, that are arranged in pairs of anodes and cathodes, made of iron (Fe) (Naje & Abbas, 2013; Rodrigues, 2019). To achieve the coagulation, a metal with positive charge is required, as it will neutralize the negative charge of dissolved and suspended particles in the water (Safe Water, n.d.). When using the EC, the electrochemical reactions needed to achieve the coagulation are induced by a direct current electric field applied to the electrodes (Naje & Abbas, 2013; Rodrigues, 2019). Therefore, the particles will attach to each other and form large agglomerates that settle to the bottom.

In this research, the EC was initially used with the aim of phosphorus recovery and water treatment. Phosphorus precipitates with iron, in the coagulation process, forming  $\text{FePO}_4$ . Hence, the EC sludge is then taken into a phosphate recovery process. Then, the remaining cleaner water, that still contains the most part of nitrogen, should flow through the next step of the treatment train.

After a period of testing running the pilot with EC, Evides decided to change it to a traditional coagulation process with iron (III) chloride ( $\text{FeCl}_3$ ). Chapter 3.0, containing the results, has a more detailed explanation for this change.

- **Chemical coagulation ( $\text{FeCl}_3$ ):** Coagulation is a conventional process, in which chemicals are added, and react with colloidal particles to form large aggregates. These, can then be more easily and rapidly removed by flocculation or membrane filtration (Oriekhova & Stoll, 2014). According to Racar et al., (2017), some conventional coagulants are ferric or aluminum salts, that enable the formed aggregates to be removed as sludge.

This process was applied in order to replace the EC unit, by dosing  $\text{FeCl}_3$ , as coagulant, with a content of Fe of 20 mg/L. Although following a different method, it was still used to precipitate phosphorus with iron and form  $\text{FePO}_4$ .

- **Anaerobic digestion (AD):** An anaerobic<sup>2</sup> digestion process consists of the degradation of natural polymers and soluble organic compounds into methane ( $\text{CH}_4$ ) and carbon dioxide ( $\text{CO}_2$ ) (Aiyuk et al., 2006) which can be used to produce electricity and heat (de Graaff et al., 2010). Besides that, the remaining part of organic matter treated by AD, known as digestate, is rich in nutrients, such as phosphorus and nitrogen, and is commonly used as an organic fertilizer in agriculture (European Biogas Association, 2015).

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<sup>2</sup> A process that occurs in the absence of oxygen.



Phosphorus that was precipitated in the coagulation process is contained in the side stream of the coagulation unit; the produced sludge. Therefore, this sludge was fed to the AD unit, which has as a by-product a digestate rich in phosphorus. Hence, the anaerobic digestion enables the recycle of this valuable nutrient from organic waste streams.

- **Nanofiltration (NF):** Is a pressure-driven membrane process, for liquid-phase separations, used to remove solutes with low molecular weight (Mulyanti & Susanto, 2018). Nanofiltration membranes have a characteristic pore size of 1 – 5 nm and its operating pressure varies between 7 – 30 bar. Therefore, its properties are classified between non-porous Reverse Osmosis (RO) membranes (where transport is governed by a solution-diffusion mechanism) and porous Ultrafiltration (UF) membranes (where separation is usually assumed to be due to size exclusion and, in some cases, charge effects) (Shon et al., 2013). Furthermore, these membranes have negative surface charge at neutral pH, which influences the rejection of cations. Consequently, both charge effects and sieving mechanisms influence the rejection behavior of solutes in NF membranes (Al-Amoudi & Lovitt, 2007; Rodrigues, 2019), making it highly selective.

This technology was applied, in the Evides pilot plant, in the nitrogen recovery route. Water that leaves the EC unit was used to feed the nanofiltration operation, aiming to remove cations, polyvalent ions, and organic matter. Their unit consists of two stages (first stage 2 membrane modules, second stage 1 membrane module). The compounds that were retained by the membrane are the concentrated phase and were returned to the sewer. Nitrogen species that permeated the membrane, flow, along with the cleaner water, to the next process unit. To achieve their goals, Evides used a polymeric membrane with a molecular weight cut off (MWCO) of 800 Da.

- **Reverse Osmosis (RO):** Reverse osmosis is a process for desalinating water using membranes that are permeable to water but essentially impermeable to salt. Different to other membrane processes, such as NF, RO membranes are dense and do not have distinct pores (Baker, 2004). This technology consists of the inverse of the natural osmosis, that means, in this process, water flows through a semi-permeable membrane from the more concentrated to the more diluted solution. For this to be possible, the applied pressure on RO, that ranges from 10 to 70 bar, must be enough so that water can be able to overcome the osmotic pressure (Shon et al., 2011; Rodrigues, 2019).

Evides applied this process for nitrogen recovery. A vertical reverse osmosis (vRO) unit was used to reject salts that were still present in the water. Nitrogen was separated from water at this unit process, as it is retained by the membrane and goes to the concentrated stream. This nutrient was later used to cultivate algae.

- **Algae:** Microalgae are photosynthetic organisms that grow rapidly and can live in harsh conditions due to their unicellular or simple multicellular structure (Barbera et al., 2018; Rodrigues, 2019). They can be used in wastewater treatment for a different range of objectives: removal of coliform bacteria, reduction of both chemical and biochemical oxygen

demand, removal of N and/or P, and also for the removal of heavy metals (Abdel-Raouf et al., 2012). Algae are also related to energy saving, since they use solar energy, because of their photosynthetic capabilities, to biodegrade organic pollutants and transform them into useful biomasses, such as biogas substrate, biofuels, fertilizers, and biopolymers (Abdel-Raouf et al., 2012; Arashiro, 2016).

An algae cultivation process was used as the final part for the nitrogen recovery route. This unit received the concentrated flow from the RO, which contained nutrients, such as nitrogen which could be used by the algae for their growth. As the algae grew, they degrade organic pollutants whilst accumulating nitrogen into their cells. Thus, it produces useful biomasses, that are rich in nitrogen.

Evides cultivated the algae *Galdieria Sulphuraria*, initially on synthetic RO concentrate. Through this process, the nitrogen residue in the wastewater was converted into the blue pigment *phycocyanine*, a valuable substance.

Furthermore, the operational parameters of some technologies of the treatment train used are presented in Appendix A, Table A1.

## 3.2 Southern Water

### 3.2.1 Process flow diagram

Figures 5 and 6 present Southern Water’s process flow diagrams for phosphorus recovery.

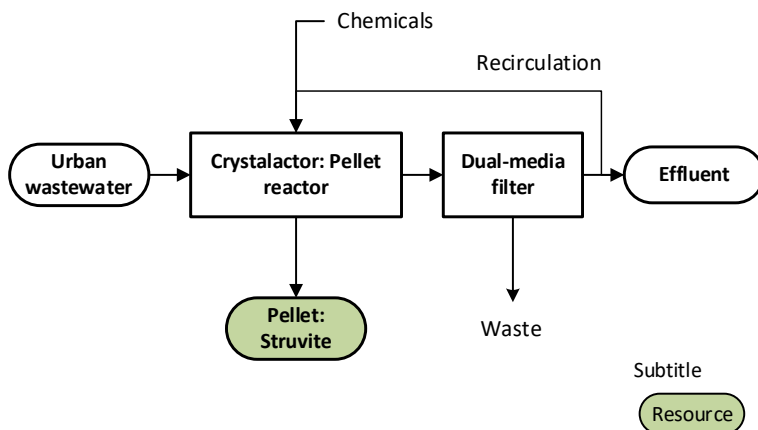


Figure 5 - Southern Water’s process flow diagram for struvite

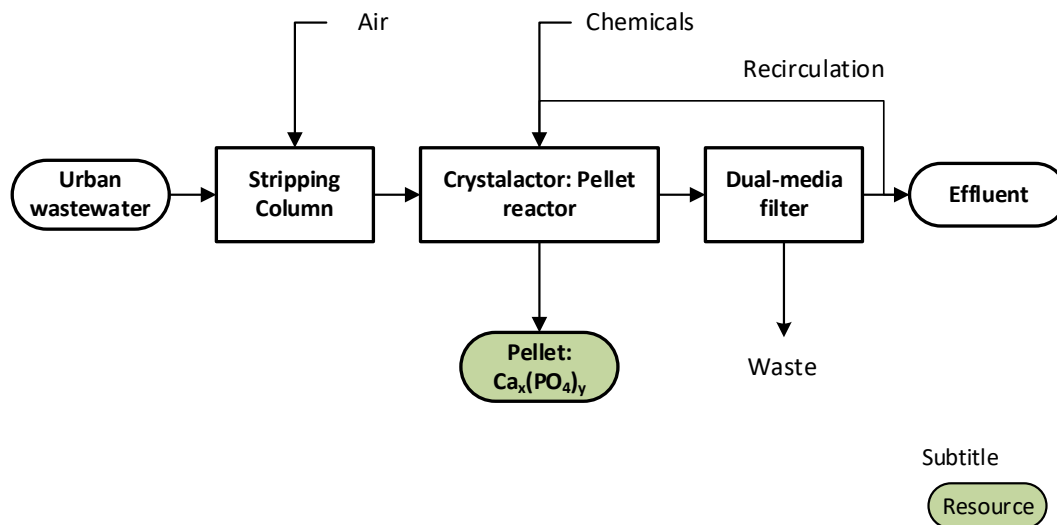


Figure 6 - Southern Water's process flow diagram for calcium phosphate

### 3.2.2 Technologies

For recovering phosphorus, Southern Water used a pellet reactor technology, named Crystalactor<sup>3</sup>, besides pre and post treatment. These processes are described below:

- Stripping column: Stripping is the process of removing gas solutes that are absorbed in a liquid, (Nguyen, 2006) by contacting it with another gas, in which the solute has more selectivity. It is governed by a phenomenon known as mass transfer, that is dependent on the relative concentrations of the gas in liquid vice-versa, and the temperature of the liquid. Therefore, to reduce the amount of the solute that is dissolved in the liquid to be treated, its temperature is increased and/or the gas concentration in contact with the liquid is reduced (VEOLIA, n.d.).

At Southern Water's pilot plant, a stripping column was used, as a pre-treatment, to remove CO<sub>2</sub> from the wastewater in the calcium phosphate route. This process was applied in order to remove carbonates, so they do not react with the calcium present in the stream to form calcium carbonate, an undesired component for this route.

- Crystalactor<sup>®</sup>: The Crystalactor is a fluidized bed type crystallization technology developed and used for selective removal and recovery of components from water and wastewater in the form of hard pellets (van Duyvenvoorde & Verhoek, 2018). Its principal is similar to the conventional precipitation; however, the transformation is controlled accurately, and the

<sup>3</sup> The Crystalactor<sup>®</sup> is the registered trademark for fluid-bed crystallizer systems developed by DHV for water treatment.

pellets have a typical size of approx. 1 mm instead of fine dispersed, microscopic sludge particles (Giesen et al., 2009).

*The heart of the Crystalactor® treatment plant is the pellet reactor partially filled with suitable seed material such as sand, small crushed pellets or minerals. The water is pumped in an upward direction, maintaining the pellet bed in a fluidized state. To crystallize the target component on the pellet bed, a driving force is created by a reagent dosage and where necessary pH-adjustment. By selecting the appropriate process conditions, co-crystallization of impurities is minimized, and high-purity crystals are obtained. The pellets grow and move towards the reactor bottom. At regular intervals, a quantity of the largest fluidized pellets is discharged from the reactor (while the reactor remains in operation) and fresh seed material is added. After atmospheric drying, easy-to-handle and virtually water-free pellets are obtained. (van Duyvenvoorde & Verhoek, 2018, p.5)*

The pellet reactor was used for both types of phosphorus recovery. For the struvite route, the reactor was fed with a sludge liquor, an addition of magnesium chloride ( $MgCl_2$ ) and sodium hydroxide (NaOH) and with seeding material (quartz sand, filter sand and filter anthracite), to enable phosphate precipitation. Calcium phosphate recovery followed a similar process as struvite; however, the reactor was fed with the stripping column effluent, and the chemicals used were chloride acid (HCl), calcium chloride ( $CaCl_2$ ) and NaOH.

- Dual media filter: This type of filter consists of two layers of different material, and can function as a progressive sieve (Zouboulis et al., 2007). The upper layer is coarser, usually made of anthracite, and is able to trap larger solids. The bottom sand layer works by trapping smaller impurities and particles (Nathanson, 2010; Zouboulis et al., 2007).

The dual media filter was used to remove amorphous material, that is, that have no detectable crystal structure, from the recirculation stream. Therefore, it separates the desired crystal (struvite or calcium phosphate), from other precipitates.

The complete unit consisted of a pellet reactor with feed pump, an operational buffer tank, dosing equipment, a stripper tower, and a dual media filter. The unit's operational parameters are presented in Appendix A, Table A2.

### 3.3 CAPSO

#### 3.3.1 Process flow diagram

Figure 7 illustrates the processes involved in CAPSO's plant for dried sludge recovery.

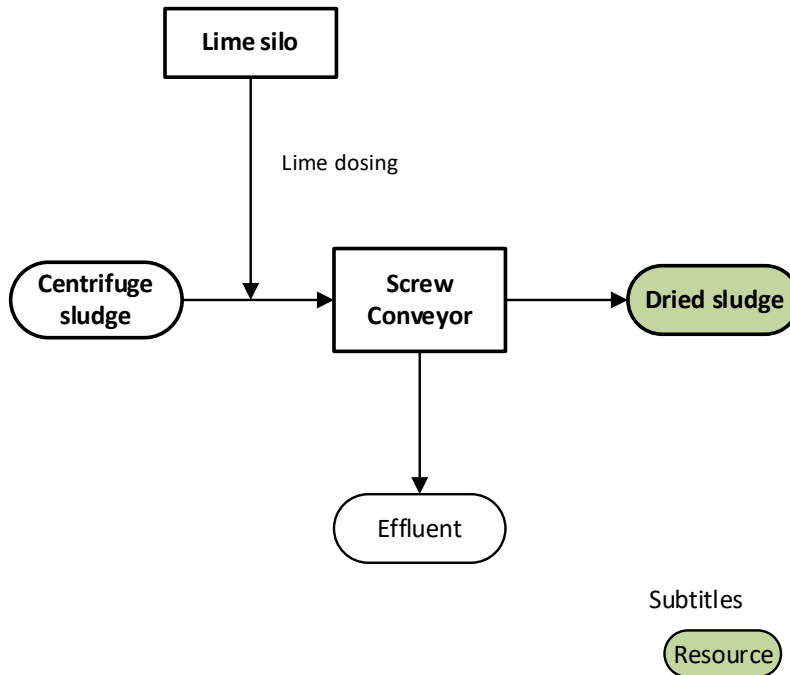


Figure 7 - CAPSO's process flow diagram

### 3.3.2 Technologies

The used technologies for recovering dried sludge, as illustrated in Figure 7, are described below:

- Lime: Lime is one of the most used amendment materials for sewage sludge stabilization at reducing pathogen content and enabling its regrowth (british lime association, 2021; Liu et al., 2012). It also increases dry solids content, which is effective in retaining heavy metal in their structure (british lime association, 2021; Judd, 2021).

Therefore, CAPSO used lime to improve the sludge's characteristics for subsequent use, converting it into a biosolid product for soil conditioning. They aimed to reduce odor and enhance its agricultural benefits by maintaining the nutrient bio-availability. The lime used was *Neutralac*<sup>®</sup> Q90SR, which is a calcium reagent.

- Screw conveyor: Screw conveyors are widely employed in industrial fields for transporting and/or mixing bulk and particulate materials at controlled and steady rates (Pezo et al., 2015).

This technology was used to mix the lime with the sludge in order to achieve the desired siccidity rate ( $\geq 30\%$ ), which is CAPSO's final product. Besides that, this type of conveyor is enclosed, providing an additional safety measure and helps with odor control.

## 4 Results

This chapter will focus on the results obtained by each pilot partner when recovering the aimed resource, as well as the recovery percentage and quality of the end product. It also presents the process optimization applied in each plant and the conditions chosen as optimal for achieving their targets.

### 4.1 Evides Industry Water

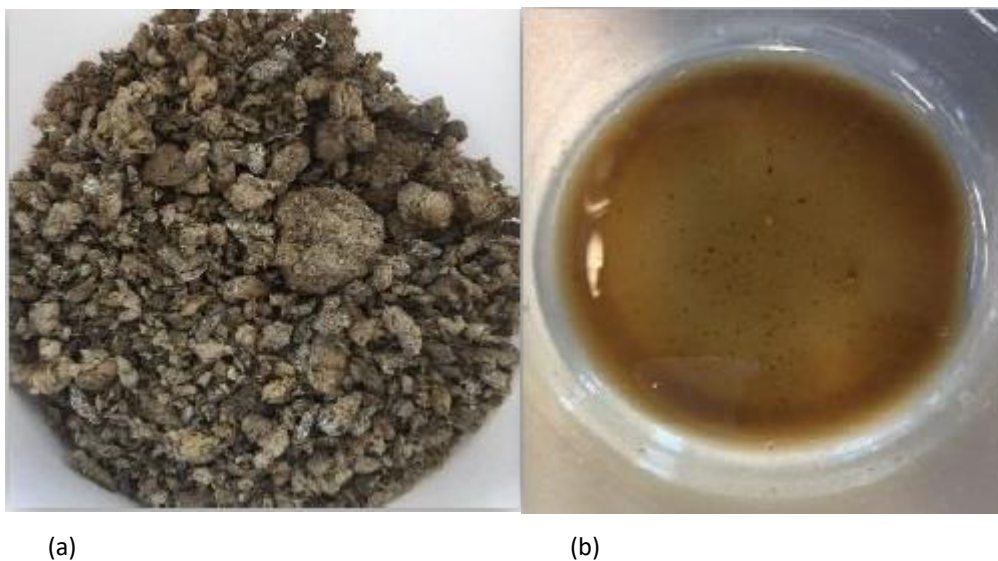
#### 4.1.1 Recovery percentage

As explained in this report, initially Evides aimed to recover, during the pilot phase, 25% of both nitrogen and phosphorus. For cellulose (C-source), there was no initial target for recovery, due to the lack of previous experience with it.

Although not working with cellulose recovery before, Evides managed to achieve a recovery rate of 65%, and convert about 39% of it into COD. Therefore, the final recovery as COD was about 27%. Similar to the C-source case, recovering nitrogen using algae was also a new process for Evides. Within the results, it was noticed that it was possible to remove all nitrogen from the stream via algae growth (100%), however, the pigment extraction can be difficult to achieve. Finally for phosphorus, an overall recovery of 31% was achieved with this treatment train. Therefore, Evides managed to exceed all of the initial goals concerning recovery.

#### 4.1.2 Recovered product

This section covers the quality of the recovered resources and its end use suitability. Cellulose, one of the resources from Evides' train, was recovered from the screenings of the sieving process, and is shown in Figure 8a. It was then converted, through the enzymatic conversion unit, to COD, a carbon source, which is illustrated in Figure 8b.

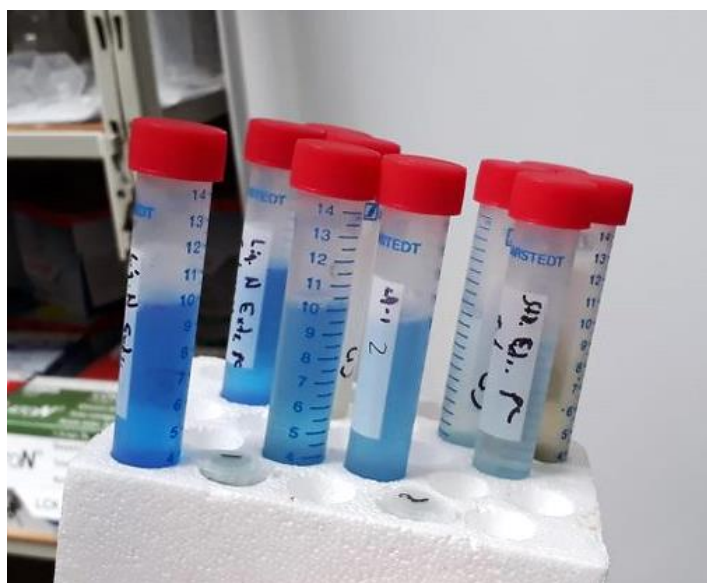


*Figure 8 - Cellulose product*

a. Cellulose; b. COD in solution

According to Evides the final product, which is the converted COD, is suitable for being directly applicable in a wastewater treatment plant. Recovering a carbon source is of high relevance, as it is needed in WWTPs that have a biological removal of nitrate, which commonly occurs in conventional treatments.

Another resource recovered by Evides was nitrogen. In effect, this nutrient was not directly recovered but used to cultivate algae, that was able to use all the nitrogen from the stream for its growth. The final product aimed for this recovery train was the pigment extracted from the algae culture, presented in Figure 9. However, according to Evides, the pigment has proven to be hard to extract, leading to the need for extra research and tests to define the feasibility of its use.



*Figure 9 - Pigment phycocyanine extracted from algae*

Another nutrient recovered in Evides' train was phosphorus, from the anaerobic digestion of the Fe-sludge (precipitation unit). Due to the change of location of the plant, as mentioned earlier in this report, Evides had no end user for the recovered products, leading to not defining a desired use for the phosphorus recovered. Nonetheless, phosphorus has proven to be an important nutrient for fertilizer, and its recovery as AD digestate enables this application since it is not lost during the digestion but concentrated.

### **4.1.3 Process optimization**

As already described in this report, Evides aimed to recover different types of resources, and, for this reason, had different trains in the complete process. For this reason, this section is divided into the optimization applied per resource.

#### **4.1.3.1 Cellulose**

In order to improve the recovery of carbon source from cellulose, Evides initially tested different enzyme dosing ratios (enzyme/dry matter), in a lab-scale, with a constant dry matter percentage of 10%. These tests were done with the screenings from the sieves with a cellulose content of 38%, and achieved a conversion efficiency, of cellulose to COD, of 36.6 – 69.4%.

The tests were then reproduced in the pilot unit, however, with a 2.5% dry matter dosage (DM) of substrate. Both lab and pilot tests results are presented in Table 5.

*Table 5 - Substrate and enzyme dosage optimization*

Scale	Substrate dosage (w/v) %	Enzyme dosage (mg/g DM)	Net Glucose (g/L)	Net COD (g/L)	Cellulose conversion %
Lab	10.0	50	25.7	41.7	69.4
Lab	10.0	20	17.7	25.3	42.2
Lab	10.0	10	9.8	21.9	36.6
Pilot	2.5	50	11.2	17.7	118.2
Pilot	2.5	20	2.4	9.0	59.9
Pilot	2.5	10	0.9	6.0	40.1

(Steenbakker, 2019)

As seen in Table 5, using a dry solids concentration of 2.5% instead of 10.0% enhanced the cellulose conversion. Also, the enzyme dosage of 50 mg/g DM gave the best results during this test. According to Steenbakker, 2019, the conversion higher than 100%, indicated the conversion of other (extra) organic material present.

After more optimization trials, Evides concluded the dosage of 2.5% substrate and 20 mg/g DM of enzyme as the optimum point. This case had the best combination, for their purpose, of cellulose conversion and external dosage.

#### 4.1.3.2 Nitrogen

An important part for the nitrogen recovery route was the use of membrane filtration, as it enables separating N from P, with the nanofiltration technology, and then N from water, with reverse osmosis.

For the nanofiltration, Evides worked with NXFiltration membranes, and tested different MWCOs (molecular weight cut-off). Figure 10 shows the retention of different solutes on membranes with MWCO of 200<sup>4</sup> Da, 400 Da and 800 Da.

<sup>4</sup> By the time the tests were done (2019), the 200 Da membranes were not according to supplier's satisfaction. Thus, a membrane was sent to the project only for testing.



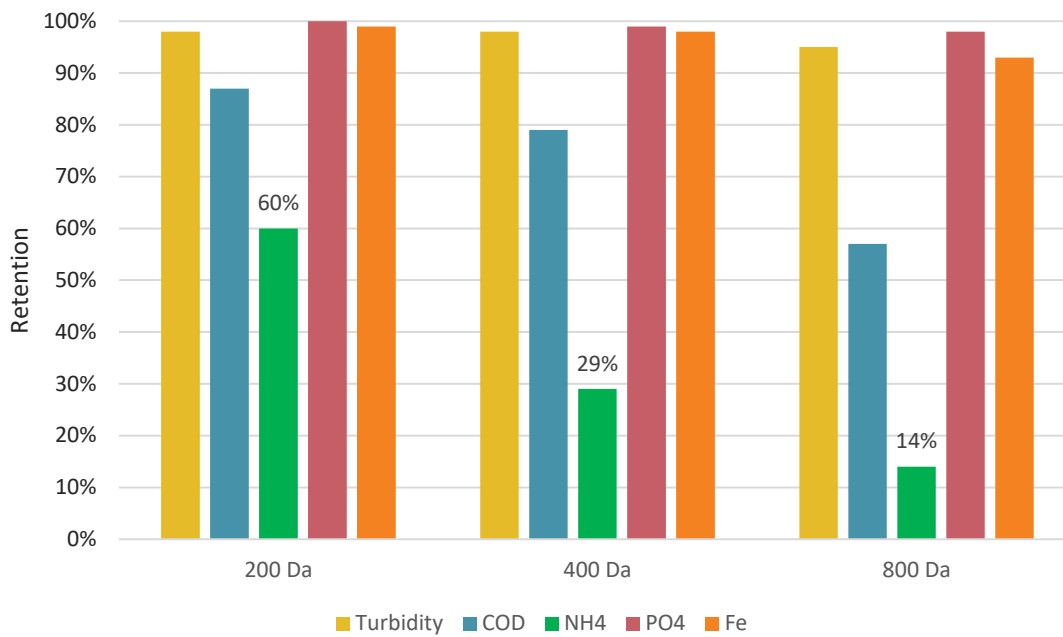


Figure 10 - Solute retention on nanofiltration membranes

As seen in Figure 10, 800 Da membrane has the lower ammonium retention (14%), therefore, enables more nitrogen passage, and its recovery on the vRO concentrate. Thus, this membrane offered the best conditions for this purpose.

As the nitrogen contained in the vRO concentrate aimed to be recovered/used by algae, a pre-study was done in order to choose the best species for cultivation. For this, they analyzed two algae species, *Galdieria Sulphuraria* and *Dunaliella salina*, based on some of their characteristics, such as: operational conditions (pH, temperature, ammonium) and the potential of monoculture.

Based on their criteria analyzes, *Galdieria Sulphuraria* was chosen to be used on the cultures. This was justified as it has an acid pH range (1-6), which facilitates the cultivation of a pure monoculture. Besides that, a value pigment, *C-Phycocyanin*, is produced by this type of algae.

With the chosen species, Evides tested different growth conditions in order to find the optimum points, such as temperature, pH, mixotrophic or autotrophic growth, etc. Figure 11, a and b, show the autotrophic and mixotrophic, with glucose addition, growth, respectively.

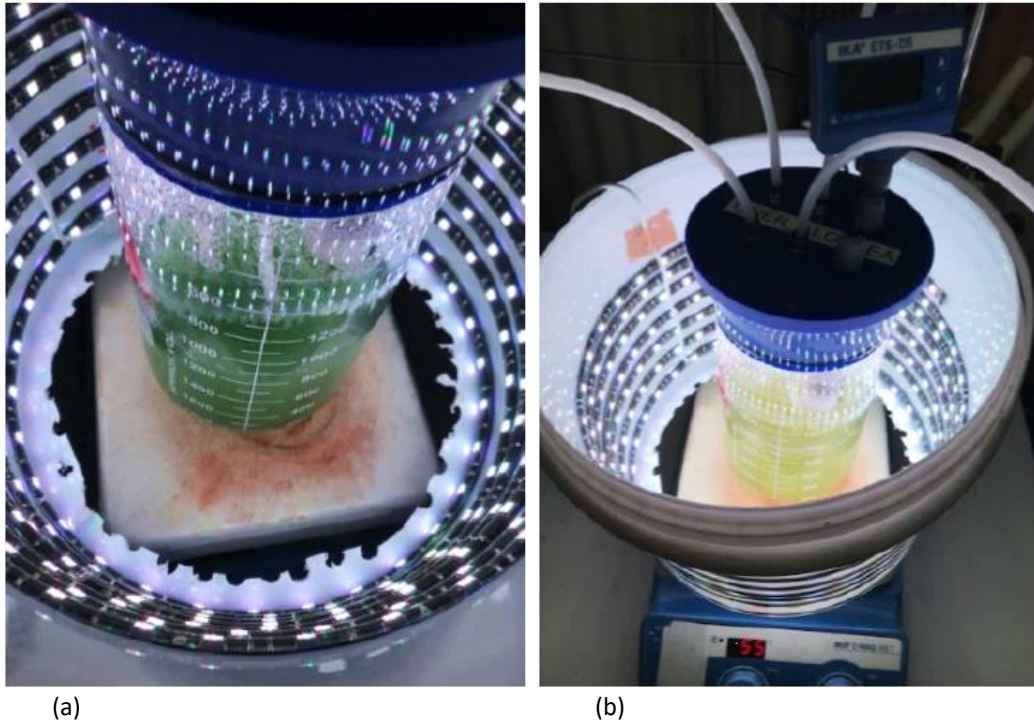


Figure 11 - Autotrophic and mixotrophic algae culture

a. autotrophic; b. mixotrophic

By concluding the trials in the pilot setup, it was noticed that the growth rates of an autotrophic proved low (0.08 u/d) when compared with mixotrophic (0.7 u/d). Therefore, mixotrophic was the best option for upscaling to a photobioreactor, shown in Figure 12.



Figure 12 - Photobioreactor for algae cultivation

### 4.1.3.3 Phosphorus

The phosphorus recovery train started with a coagulation process; initially iron electro-coagulation, that was later replaced by a coagulation with iron chloride dosing. The optimization procedure for both is explained in this section, and also the reasons that led to the replacement of the technology used.

The first tests concerning the EC-pilot consisted of applying different amperages, varying between 200 – 800 A. The results indicated that higher amperage resulted in a better coagulation and turbidity removal, however, not appropriate for running the treatment train (Steenbakker, 2019). It was concluded that the flocculation vessel, which is part of the EC system, almost did not contribute for the settling and removal of particulates. Therefore, new lab tests were done (jar-test) with a slow mixing of the flocculation tank, which improved the turbidity removal. The jar-test experiments presented results up to a turbidity removal of 80%, particulate COD of 90% and total phosphorus (TP) of 90% (van den Brink & van de Griek, 2019).

Although the initial improvements achieved in the particulate removal in the lab-scale, these results were not achieved in the pilot setup. Additionally, Evides faced challenges with iron precipitates accumulating inside the bottom of the EC reactor, on the NF membrane and also in its permeate. Some hypotheses were that the oxidation of Fe(II) to Fe(III) was not sufficient, the flocculation and settling were not optimal and the design of the EC reactor.

After some optimization trials, such as changing the aeration rate and the reactor design, Evides decided to replace the EC unit for a coagulation reactor with FeCl<sub>3</sub> dosing. The optimum iron dosing was found to be 20 mg/L, as higher dosages did not add extra benefit.

Succeeding the definition of the best conditions for the coagulation unit, the recovery of phosphorus, as PO<sub>4</sub>, in the AD digestate was also studied. The effect of increasing the pH with a solution of 32% NaOH was investigated, by dosing between none to 20 mL/L of digestate. It was noticed that increasing the pH from 7.5 to 12.4 increased the recovery from 31% to 39%, respectively. Although the improvement with higher pH, it was not feasible due to cost of chemicals. Therefore, it was decided to not control the pH, as it already presented a P-recovery.

### 4.1.4 Full scale design

According to Steenbakker & van den Brink, 2021, the plant should be at full scale in order to be economically feasible. It was concluded that resource recovery can reach a full-scale design depending on the stream, source and application. However, it is not intended to go full-scale with this same setup

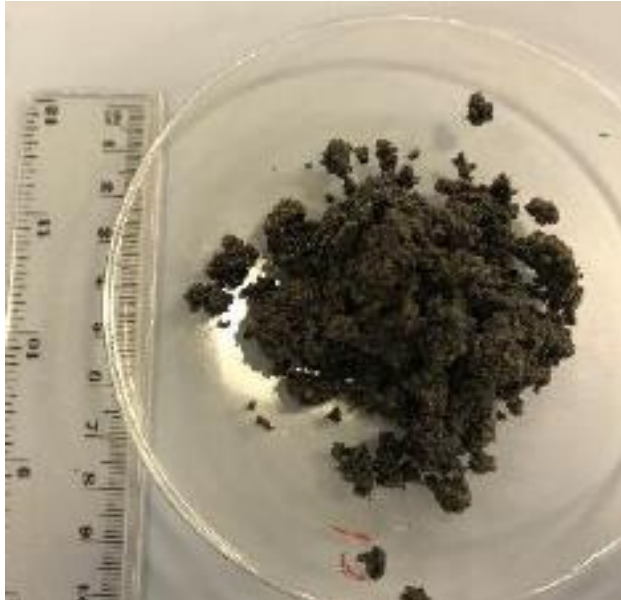
## 4.2 Southern Water

### 4.2.1 Recovery percentage

The treatment train used for recovering struvite achieved a maximum phosphorus removal of 95% from the wastewater, at a pH of 8.5 (Randall & Hossain, 2021), of which an average of 78% was precipitated. Concerning the calcium phosphate precipitation, this process also removed about 95% of phosphorus, at pH 9.12 (Randall & Hossain, 2021), of which an average of 93% precipitated.

#### 4.2.2 Recovered product

Southern Water recovered phosphorus through two precipitates: struvite and calcium phosphate. A picture of the struvite product is showed in Figure 13, and its composition is available in Table 6.



*Figure 13 - Struvite product*

(Randall & Hossain, 2021)

*Table 6 - Struvite composition*

Element	Concentration	
	mg/kg DM	% w/w
Aluminium	1.32	0.4
Calcium	20.73	6.1
Iron	2.02	0.6
Magnesium	13.32	3.9
Phosphorous	31.94	9.4

*Note.* Modified from (University of Portsmouth Higher Education Corporation, 2019)

According to Huang et al., 2010, a theoretical pure struvite has in its (mass) composition 9.9% of Mg, 5.7% of  $\text{NH}_4\text{-N}$  and 12.6% of  $\text{PO}_4\text{-P}$ . The composition of the pellet seems reasonable for phosphorus; however, it is much lower for magnesium and with a high impurity as calcium. Also, according to Southern Water’s report, ammonium values were very low (van Lit & Gerard, 2019).

Calcium is known for having an impact on struvite crystal size, shape and purity as it interacts effectively with phosphate (Le Corre et al., 2005). This could result in part of the phosphate reacting with calcium instead of magnesium, explaining the found composition. Therefore, it was found that it was not conclusive which phosphate salts were formed during the struvite route.

The calcium present could also be a cause for the stability of the pellet, that has been reported as fragile and small, and sometimes without the desired crystal (Randall & Hossain, 2021). According to Le Corre et al., 2005, the increase of calcium concentration reduces the crystal size or can lead to a

formation of an amorphous calcium phosphate. Another reason for this, could be the amount of sand in the pellet, that was about 66%, and should be of 3% - 5% for full-grown pellets (van Lit & Gerard, 2019). Randall & Hossain, 2021, from Southern Water, reported that due to on-site regulations the pilot could not run continuously and therefore had to be shut down and re-started every day, which likely caused interference in the crystal growth.

Figure 14 presents a picture of the calcium phosphate product, and Table 7 shows its composition.



*Figure 14 - Calcium phosphate product*

(Randall & Hossain, 2021)

*Table 7 - Calcium phosphate composition*

Element	Concentration	
	mg/kg DM	% w/w
Aluminium	0,21	0,1%
Calcium	28,75	8,1%
Iron	2,59	0,7%
Magnesium	0,51	0,1%
Phosphorous	21,42	6,0%

*Note.* Modified from (University of Portsmouth Higher Education Corporation, 2019)

Based on the composition presented in the Table 7, calcium phosphate had a Ca/P ratio of about 1:1, which indicates that some calcium likely reacted with carbonate that was still present.

Southern Water reported the same issues with the pellets that occurred with struvite; fragile and small, and with a sand concentration of about 64% (Randall & Hossain, 2021). The consequences of having to re-start the pilot every day, as explained before, is also accounted for here.

#### **4.2.3 Process optimization**

In order to obtain a representative testing of the Crystalactor pilot and the best conditions for achieving the end product, two trial routes were conducted on the plant; calcium phosphate and struvite production.

The trial for the struvite phase consisted of the following objectives (van Duyvenvoorde & Verhoek, 2018):

- Determine adequate chemical process condition to obtain stable crystallization for struvite. The parameters investigated for this were NH<sub>4</sub> concentration, pH, crystallization efficiency, crystal growth and strength, pre-treatment/filtration needs;
- Optimize the reactor load by changing the recirculation ratio;
- Optimize chemical process conditions (Mg dosage, NH<sub>4</sub> concentration, pH);
- Test different types of seeding materials.

Table 8 presents some of the conditions tested, for struvite precipitation, and their respective results. The P removal column accounts for the total removal of phosphorus from the feed stream, whilst the P crystalized is the amount of phosphorus removed that actually crystalized into struvite.

*Table 8 - Optimization conditions and results for struvite*

Condition	P removal (%)	P crystalized (%)
Initial operation and 50% MgCl <sub>2</sub> dosing	82	66
No MgCl <sub>2</sub> dosing and pH 8.0	79	69
Excess MgCl <sub>2</sub> dosing and pH 7.5	76	49
No MgCl <sub>2</sub> dosing and pH > 8	88	50
Slight excess MgCl <sub>2</sub> dosing and pH > 8	91	78

Therefore, the optimum conditions, between those tested, for struvite precipitation included a high pH with a slight Mg excess. NH<sub>4</sub> excess, that was also tested, did not have a positive effect on the crystallization efficiency.

For the calcium phosphate route, the optimization tests were similar to struvite, but with conditions adapted for this type of precipitate (van Duyvenvoorde & Verhoek, 2018):

- Determine adequate chemical process condition to obtain stable crystallization. This accounted for the investigation of pH, crystallization efficiency, crystal growth and strength, pre-treatment/filtration needs;
- Optimize the reactor load by changing the recirculation ratio;
- Optimize chemical process conditions (Ca/P-dosage, pH, concentration of carbonate in feed);
- Test different types of seeding materials.

Table 9 presents optimization tests done for the calcium phosphate route and the achieved results. Calcium excess is in mmol per liter of influent feed.

Table 9 - Optimization conditions and results for calcium phosphate

Condition	P removal (%)	P crystalized (%)
Ca excess 6.5 and pH 6.5	75.0	52.3
Ca excess 6.5 and pH 8.5	87.5	90.0
Ca excess 6.5 and pH 9.2	96.5	93.8
Ca excess 9.4 and pH 7.2	76.9	67.3
Ca excess 13.5 and pH 7.2	77.8	62.2

With the optimization tests, it was concluded that a higher pH led to both higher removal and crystallization of phosphorus. Concerning the addition of calcium, it only led to an increase from 52% to 62% in P crystallization, and therefore, was considered not relevant for the process.

#### 4.2.4 Full scale design

According to Southern Water, there are no current plans to go full scale with this plant as data showed that it would not necessarily be cost effective and whilst other benefits need to be further researched. They also have other areas of the business looking at nutrient recovery and how to best manage their bio resources. However, their current focus is with water recycling in order to address their needs

### 4.3 CAPSO

#### 4.3.1 Recovery percentage

As CAPSO's project was to dewater sludge, it only removes water from it and recovers all the influent solids, meaning about 100% recovery of the sludge valuable products; e.g., nutrients. Therefore, a better comparison for this scope is to consider the amount of water removed and their dryness goal.

The influent sludge had a siccidity of 25%, which means that the sludge was made up of 75% of water. The end product sludge produced at the end of the pilot had a siccidity of 32%, that is, 68% water content. This means that the overall de-watering achieved meant a reduction in water content of 7%. This removal enabled CAPSO to get to their dryness rate goal; above 30% siccidity. This is further discussed in *section 4.3.2*.

#### 4.3.2 Recovered product

CAPSO recovered sludge with an average siccidity level of 32% and stack height > 1.80m. According to CAPSO, 2020, this rate of dryness is equivalent to a rate that is reached with press filters, which are more efficient dewatering equipment but also much more expensive, both in investment and operation.

Besides that, concerning regulations in France, by reaching a dryness level of  $\geq 30\%$ , that is water  $\leq 70\%$ , they are no longer obliged to cover the sludge storage area (e.g. build a warehouse), and are able to save costs. Also, the dried sludge is easier to transport.

Concerning the end sludge quality, the composition of organic and mineral material of the sludge, important factors for the use as fertilizer, are presented on Table 10. According to Monsterleet, C., from CAPSO, these parameters do not have a regulatory limit, and are therefore analysed to define the agronomic value of the sludge and thus calculate the maximum amount of sludge that is

necessary to add to the fields. However, for metallic trace elements and organic trace elements there are regulatory thresholds set by law, for which the dried sludge has to comply. Figure 15 shows the distribution of these elements in the sludge accordingly to the law limits, that is, considering the limits are 100% on the graph, it presents the ratio between the sludge composition and the limit value.

Table 10 - Dried sludge composition

Parameter	Unit	Value
pH	-	12.4
Organic material	%	12.0
Mineral material	%	14.8
Total nitrogen	g/kg	11.6
Organic nitrogen	g/kg	11.6
Organic carbon	%	6.0
Total phosphorous (P <sub>2</sub> O <sub>5</sub> )	g/kg	13.6
Total potassium (K <sub>2</sub> O)	g/kg	1.5
Total Magnesium (MgO)	g/kg	1.5
Total Calcium (CaO)	g/kg	78.4
Sodium (Na <sub>2</sub> O)	g/kg	0.29

(Dupont, 2017)

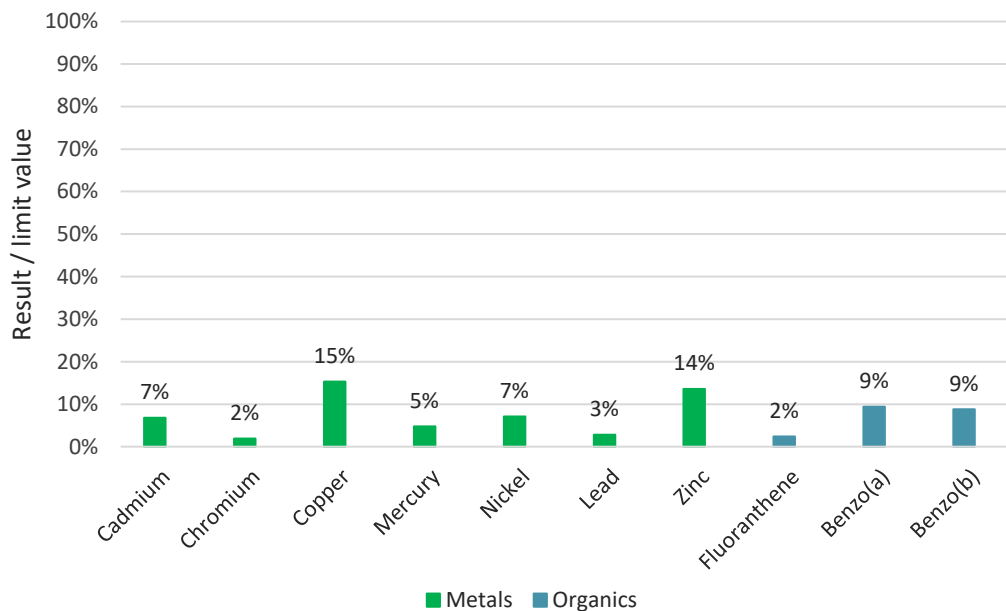


Figure 15 - Metallic and organic trace elements compared to the law limits

Note. Adapted from Dupont, 2017

As seen in Figure 15 metal and organic trace elements in the sludge are below limits and complying with the regulation, enabling its end use. The final product is then meant to be applied as a soil conditioner by local farmers; the added lime, and the nutrients contained in the sludge can increase soil fertility, allowing the increase of crop yields (CAPSO, 2020a). Also, it is easier to be spread mechanically on the fields, due to the achieved dryness level.

Figure 16 shows CAPSO's end product: dried sludge.





*Figure 16 - Dried sludge for soil conditioning*

#### **4.3.3 Process optimization**

In order to meet their targets, CAPSO applied modifications to the sludge process at the Saint-Omer wastewater treatment plant, by replacing equipment and testing different types of limes. Initially, they focused on the technologies that were used on the site, and replaced the feeder pump and its discharge by the installation of a lime mixer and three screw conveyors.

They also tested three different types of limes (Neutralac<sup>®</sup>: Q3, Q200HR, Q90SR) at different dosing rates (20%, 25%, 30% and 35%). It was concluded that Q90SR lime, dosed at 35%, was the best condition to meet the regulatory targets (CAPSO, 2020b).

The sludge recovery system before this study (centrifuges) was only able to achieve a dryness level of around 25% and stack height lower than 1.50m. With the modifications done on the site and the optimization tests of the lime dosing, they were able to meet their targets, by achieving a siccidity of 32% and a stack height higher than 1.80 m.

#### **4.3.4 Full scale design**

The current plant is already full scale, and according to CAPSO, 2020a, it yielded about 979 tons of dry sludge in 2019. Additionally, this demo case can be applied in any plant with the right lime dosage, as influent quality changes per location and type of wastewater treatment plant (Courouble, 2021).

## 5 Discussion

During this report, characteristics of the resources being recovered, along with the technologies used and the obtained results have been presented per pilot partner. In order to enable a discussion about all of these recovery processes, this chapter will focus on comparing the main aspects and results of their processes; their technologies, recovered product and achievement of initial goals and targets.

### 5.1 Technologies and processes conditions

As all the technologies involved have already been presented per pilot partner in *chapter 3*, the aim here is to provide a better view and comparison of them. Therefore, Table 11 presents the overall and most relevant process conditions that have been presented in this report.

*Table 11 - Overall process overview per recovered resource*

	Evides			Southern Water		CAPSO
	C-source	Phosphorus	Nitrogen (Algae pigment)	Phosphorus and Nitrogen (struvite)	Phosphorus (Ca <sub>x</sub> (PO <sub>4</sub> ) <sub>y</sub> )	Dried sludge
<b>Main technologies</b>	<ul style="list-style-type: none"> <li>• Enzymatic conversion</li> </ul>	<ul style="list-style-type: none"> <li>• Coagulation</li> <li>• Digestion</li> </ul>	<ul style="list-style-type: none"> <li>• Membranes</li> <li>• Algae</li> </ul>	<ul style="list-style-type: none"> <li>• Crystalactor</li> </ul>	<ul style="list-style-type: none"> <li>• Crystalactor</li> </ul>	<ul style="list-style-type: none"> <li>• Screw conveyor</li> </ul>
<b>External dosing</b>	<ul style="list-style-type: none"> <li>• Enzyme</li> </ul>	<ul style="list-style-type: none"> <li>• FeCl<sub>3</sub></li> </ul>	<ul style="list-style-type: none"> <li>• pH control</li> </ul>	<ul style="list-style-type: none"> <li>• MgCl<sub>2</sub></li> <li>• NaOH</li> </ul>	<ul style="list-style-type: none"> <li>• CaCl<sub>2</sub></li> <li>• NaOH</li> </ul>	<ul style="list-style-type: none"> <li>• Lime (CaO)</li> </ul>
<b>Operational variables</b>	<ul style="list-style-type: none"> <li>• Temperature</li> </ul>		<ul style="list-style-type: none"> <li>• Pressure</li> <li>• Temperature</li> </ul>	<ul style="list-style-type: none"> <li>• Flow</li> <li>• pH</li> </ul>	<ul style="list-style-type: none"> <li>• Flow</li> <li>• pH</li> </ul>	<ul style="list-style-type: none"> <li>• Mixing</li> </ul>
<b>Barriers</b>	2	4	5	2	3	2

As pilot partners focused on recovering different resources, a direct comparison between the treatment trains is not possible. However, it is valuable to see that different types of resources can be recovered from different waste streams, and using different technologies.

As presented in Table 11, these different technologies and end products also require specific operational conditions. For example, nitrogen was recovered by Evides with the use of algae, with prior separation with membrane technologies (NF and RO). Algae needs heat and also light to grow. Nitrogen was also recovered as struvite, by Southern Water, with the Crystalactor. For this process, pH adjustment and Mg dosing, to provide appropriate Mg/P ratio is important. It is also noticeable that the feed and recirculation flow are crucial for having the correct concentration for the crystallization reaction, and also to keep the crystals inside the reactor.

The “barrier” row is meant to analyse the complexity of the treatment train, as it means the amount of units necessary to get to the end product. It is important to note that Evides treated raw domestic wastewater, needing more unit processes, whilst the influent at Southern Water and CAPSO was a by-product of a WWTP process; centrate and sludge from centrifuge, respectively.

## 5.2 Recovery percentages

Table 12 presents the recovery percentage achieved for each resource by the pilot partners. It considered how much of the resource there was in the influent stream and how much of it constituted their end product, in mass.

*Table 12 - Resource recovery percentage per pilot partner*

Cellulose (C source)	Evides		Southern Water		CAPSO
	Phosphorus	Nitrogen (Algae)	P & N (struvite)	Phosphorus (Ca <sub>x</sub> (PO <sub>4</sub> ) <sub>y</sub> )	Dried sludge
65% 32% to COD	31%	100%	P: 74.10% N: 8.00%	90.52%	100% 32% dryness

Two of the resources are presented as up to 100% recovery. For the dried sludge, as explained before, CAPSO focused on dewatering the sludge, therefore only water was removed and the amount of solids remained the same. Therefore, its dryness level is more relevant for the evaluation. Concerning the nitrogen, it could be completely removed from the water and used by the algae. Thus, this recovery concerns the nutrient taken by the algae, but does not represent the amount extracted as pigment.

## 5.3 Quality of recovered products

Table 13 contains the quality of the resources recovered at the pilot plants. It aims to provide the characteristics of the products in order to discuss whether it is suitable or not for the end use.

*Table 13 - Quality of recovered products per pilot partner*

	Evides			Southern Water		CAPSO
	C-source	Phosphorus	Nitrogen	P & N (struvite)	Phosphorus (Ca <sub>x</sub> (PO <sub>4</sub> ) <sub>y</sub> )	Dried sludge
<b>Overall aspect</b>	<ul style="list-style-type: none"> <li>• COD in solution</li> </ul>	<ul style="list-style-type: none"> <li>• Phosphate in AD digestate</li> <li>• Leaching</li> </ul>	<ul style="list-style-type: none"> <li>• Algae + pigment</li> </ul>	<ul style="list-style-type: none"> <li>• High in phosphate;</li> <li>• Fragile and small</li> </ul>	<ul style="list-style-type: none"> <li>• High in phosphate;</li> <li>• Fragile and small</li> </ul>	<ul style="list-style-type: none"> <li>• Siccidity &gt; 30%</li> <li>• Stack height &gt;1.80 m</li> </ul>
<b>Impurities</b>	<ul style="list-style-type: none"> <li>• n.a.<sup>a</sup></li> </ul>	<ul style="list-style-type: none"> <li>• n.a.<sup>a</sup></li> </ul>	<ul style="list-style-type: none"> <li>• n.a.<sup>a</sup></li> </ul>	<ul style="list-style-type: none"> <li>• Yes, some metals</li> </ul>	<ul style="list-style-type: none"> <li>• Yes, some metals</li> </ul>	<ul style="list-style-type: none"> <li>• Yes, below law limit</li> </ul>
<b>Suitable for end use</b>	<ul style="list-style-type: none"> <li>• Yes, as C-source in WWTP</li> </ul>	<ul style="list-style-type: none"> <li>• No end use defined</li> </ul>	<ul style="list-style-type: none"> <li>• Partially</li> </ul>	<ul style="list-style-type: none"> <li>• Conditional</li> </ul>	<ul style="list-style-type: none"> <li>• Conditional</li> </ul>	<ul style="list-style-type: none"> <li>• Yes, soil conditioner</li> </ul>

<sup>a</sup> n.a: not available.

As seen in Table 13, the applicability and suitability vary per resource. The COD, from cellulose, recovered by Evides, and the dried sludge, recovered by CAPSO, are suitable for their end use. Thus, directly applicable as a carbon source in WWTP and as a soil conditioner in fields for agriculture, respectively.

Nitrogen is listed as partially suitable because of the two goals involving this nutrient: algae growth and the pigment production/extraction. Nitrogen could be completely taken (recovered) by the algae, for its growth, however, the pigment extraction is not as its optimum point. Therefore, algae is a suitable end product but not its pigment.

Phosphorus was recovered by both Evides and Southern water. As they had different goals for their trains, the technologies chosen were different, thus having a direct impact on the aspect of the end products. Evides' final phosphate product is concentrated in the anaerobic digestion digestate (sludge + water), whilst SW products are solid pellets with low water content.

The pellets recovered by Southern Water were initially meant to be reused as fertilizers, as they are rich in phosphate. However, according to SW, the final application of the pellets is conditional to the approvability of farmers, which was considered to be difficult. Another challenge faced, is that currently there are no legislation, in the UK, for the use of recovered struvite and calcium phosphate as fertilizers.

#### 5.4 Initial goals and targets

An assessment of the pilot plants' end products was done by comparing the initial goals and recovery targets with the achieved ones. This assessment is presented in the form of a table containing a color coded conclusion, per recovery percentage and other goals. Table 14 contains this color code definition, and the assessment is covered in Table 15.

Table 14 - Color code definition










Color	Definition
	<ul style="list-style-type: none"> <li>Recovery target achieved</li> <li>Goals achieved</li> </ul>
	<ul style="list-style-type: none"> <li>At least 50% of recovery target achieved</li> <li>Goals partially achieved</li> </ul>
	<ul style="list-style-type: none"> <li>Less than 50% of the recovery target achieved</li> <li>None (or almost) of goals achieved</li> </ul>

Table 15 - Comparison of the initial and achieved targets and goals

	Evides			Southern Water		CAPSO
	C-source	Phosphorus	Nitrogen	Phosphorus (struvite)	Phosphorus (Ca <sub>x</sub> (PO <sub>4</sub> ) <sub>y</sub> )	Dried sludge
<b>Recovery target</b>	-	25%	25%	-	-	≥ 30% dryness
<b>Achieved</b>	<b>21%</b>	<b>31%</b>	<b>100%</b>	<b>74%</b>	<b>91%</b>	<b>32%</b>
<b>Goals</b>	Cellulose into COD	P recovery from sludge	Grow algae and extract pigment	Recover P as struvite pellets	Recover P as calcium phosphate pellets	≥ 30% dryness and ≥ 1.8 m stack
<b>Achieved</b>						

As seen in Table 15, all partners were able to achieve their recovery percentage targets, as already discussed during the report. Focusing on their goals beside the amount recovered, Evides successfully recovered cellulose from the wastewater and converted it into COD, by enzymatic conversion, and phosphorus from the anaerobic digestion residues (digestate). Concerning nitrogen, the goal was partially achieved, as they manage to grow an algae culture that was able to take up to all the N present, but the pigment extraction has proven to be difficult and not yet successful.

Southern Water achieved the goal of recovering phosphorus as calcium phosphate pellets. For the struvite route, the pellets had a phosphorus composition relatively close to the expected, however, still presented impurities, meaning that P also reacted to form other precipitates. It also did not had the expected amount of magnesium and ammonium, which led them to define the pellet as inconclusive in terms of what phosphate salts were formed. They also reported that the pellets had around 65% of sand, causing them to be fragile. Therefore, the goal was partially achieved for both pellets.

Finally, CAPSO proposed to obtain a sludge dryness ratio over 30% and a stack height of  $\geq 1.8\text{m}$ . They successfully achieved their goals, enabling the sludge to be reused as a soil conditioner by local farmers, with easier transportation and storage, due to the achieved siccidity.

## 6 Conclusion

This report covered the experiences on recovering different resources from wastewater, during the NEREUS project, by the pilot partners involved: Evides, Southern Water and CAPSO. These resources consisted of cellulose, dried sludge for soil application and nutrients; nitrogen and phosphorus. The diversity of the desired products and goals led to pilot plants varying in size and also in a broad use of technologies. These involved several trials and optimization tests in order to find appropriate conditions for recovering products with high quality suitable for re-use.

By the methodology applied by each partner, it was possible to conclude that the amount and type of resources being recovered directly influences the size of the pilot plant. Evides focused on recovering all three central products involved: resources, water and energy, in which the resources concerned phosphorus, nitrogen and cellulose. Because of this, Evides' plant was the biggest of them all and involved several technologies, whilst the other plants were more compact. Southern Water had two different products, both aiming for the recovery of phosphorus in the form of crystals, but shared one main technology for their recovery. CAPSO focused on one main resource, and for this reason, a simpler setup, but that enabled a successful recovery of sludge as a soil conditioner.

Therefore, with this information and the results achieved at each plant, it's noticeable that recovering more products requires more process units and also faces more challenges and difficulties when trying to achieve goals. However, the aim to recover more products can lead to more reuse opportunities. On the other hand, a compact plant can be easier to operate and might increase the chances of a successful recovery.

Thus, this report aimed to highlight the importance and possibilities of using wastewater as a source of products; finite resources constituted in the waste stream can be reapplied in a meaningful manner, both on the site itself or by other local users. As presented and discussed, several products can be obtained from different sources and for diverse applications. The results achieved by the partners corroborate this, by demonstrating the quantity and quality of their end products, and also their applicability. Nevertheless, it is also important to note that the path for achieving a circular economy is important but also challenging, and further research is needed in order to improve the processes, the feasibility and acceptability of resource recovery.

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## Appendix A

### Pilot plants operational conditions

*Table A1 – Evides’ pilot plant operational conditions*

<b>Technology/Input</b>	<b>Parameter</b>	<b>Value</b>
Pilot influent	Flow rate (m <sup>3</sup> /h)	2.5
Drum sieves	Supplier and type	Toro Defender
	Pore size (mm)	416
Electro-coagulation	Amperage (A)	0.25
	MWCO (Da)	150-1400
Nanofiltration	Supplier	800
	Pressure (bar)	NXfiltration
Cellulose to C-source	Dry matter dosage (%)	3.0
	Enzyme dosage (mg/g DM)	2.5
Vertical reverse osmosis	Supplier and type	50
	Surface area	DOW LE4040
Algae	Type	7.2 m <sup>2</sup>
		<i>Galdieria</i> <i>Sulphuraria</i>

Steenbakker (2019)

*Table A2 – Southern Water’s pilot plant operational conditions*

<b>Parameter</b>	<b>Value</b>
pH range feed water	6.0 – 7.0
pH after stripping tower	< 4.0
pH range effluent reactor	7 – 11
Feed flow (L/h)	8- 24
Recirculation flow (L/h)	72 – 56
Temperature	Ambient

van Duyvenvoorde & Verhoek, 2018



# Interreg EUROPEAN UNION

## 2 Seas Mers Zeeën

### NEREUS

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