PILOT STUDY ON ADVANCED PHOSPHORUS REMOVAL FROM WWTP EFFLUENT USING BIOPHREE® TECHNOLOGY

REPORT

2026

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REPORT 2026 14

ISBN 978.94.6479.133.4

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STOWA publications can be ordered at www.stowa.nl/publicaties

COLOPHON

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STOWA STOWA 2026-14
ISBN 978.94.6479.133.4

The project executors of all IPMV pilot studies have been involved in this evaluation multiple times. Throughout the duration of the program, Mirabella Mulder was the coordinator of the IPMV program.

FOREWORD

PILOT STUDY SHOWS THAT LOW EFFLUENT PHOSPHORUS LEVELS ARE ACHIEVABLE WITH BIOPHREE® TECHNOLOGY

Despite an average phosphorus removal efficiency of nearly 90% at Dutch wastewater treatment plants, the surface water in many water bodies still does not meet the requirements of the European Water Framework Directive. Phosphorus concentrations are frequently too high, and further phosphorus removal from wastewater will contribute to improved water quality.

The conventional solution for advanced phosphorus removal is the use of a posttreatment sand or cloth media filter combined with coagulant (iron chloride) dosing, which can achieve approximately 0.25 mg P/L. In this STOWA project, the BiOPhree® technology was tested at pilot scale — a phosphorus removal technology capable of achieving a phosphorus concentration of 0.1 mg P/L.

The superior effluent quality, combined with low costs and low chemical consumption, as well as the potential for phosphorus recovery, makes BiOPhree an attractive posttreatment technology for wastewater treatment plants facing strict phosphorus limits under the European Water Framework Directive. The positive pilot results provide sufficient confidence to demonstrate the technology at full scale. Several Water Authorities are currently considering a demonstration project using this technology.

Mark van der Werf
Director STOWA

SUMMARY

From October 2023 to December 2024, the BiOPhree® technology designed to meet ultra-low Total Phosphorus (TP) discharge consent limits in effluent was tested on a pilot scale at the Dronten WWTP. BiOPhree is based on phosphate adsorption to iron granlues. The technology was developed by the company Aquacare Europe BV for the treatment of cooling water for the prevention of biofouling. Since 2020, Aquacare has been working with Haskoning on the further development of BiOPhree for meeting the lowest phosphate standard for WWTP effluent.

HOW BIOPHREE WORKS

The effluent is first filtered for removal of suspended solids and is then passed over an adsorption column in which the dissolved phosphate is extensively remo-ved to < 0.1 mg P/l. Over time, the adsorbent (the iron granules) is saturated and regeneration is needed to desorb the removed phosphate. This is done with caustic soda. After regeneration, the adsorbent is again suitable for phosphate ad-sorption in a subsequent cycle.

The caustic soda released during regeneration (the regeneration) is treated with a nanofiltration membrane to maximise caustic soda recovery and to further concentrate the desorbed phosphate. The recovered caustic soda is reused in the BiOPhree process in a subsequent regeneration. The concentrated phosphate can be precipitated with calcium into hydroxyapatite or processed in a struvite reactor. The most optimal route for P recovery must be examined for each location.

RESEARCH OBJECTIVE

The research at the Dronten WWTP had two main goals:

- Demonstrate that extensive P removal from effluent is possible after multiple regenerations of the adsorbent.
- To determine the extent to which caustic soda recovery is possible from the regene-ration using nanofiltration.

TRIAL DESIGN

Pilot installation

The pilot consisted of 3 parallel adsorption columns with 1 upstream sand filter. In addition, the installation was equipped with a caustic soda tank for regenera-tion. The capacity of the pilot was 3 m^3/hour with a maximum of two columns (each 1.5 m^3/hour) in operation at the same time and 1 column on standby.

The pilot was operated by an intern who sampled the supply and discharge and took phosphate measurements on a daily basis. Regenerations were carried out by the trainee and an employee of Haskoning.

Membrane test

The recovery of caustic soda from the regeneration that was collected during the regenerations has been investigated in collaboration with EMI-Twente. To this end, a batch of regenerate was transported to a pilot location in Enschede where the effect of nanofiltration on the regenerate was tested.

Lab tests

Lab tests were carried out to gain more insight into a number of aspects:

- the regeneration strategy;
- phosphate precipitation from the regenerate;
- combined removal of suspended matter and phosphate in the adsorption column.

RESULTS

The pilot trial showed that P-removal to consistently achieve <0.1 mg P/l was possible from effluent from the Dronten WWTP that contained an average of 0.9 mg P/l. Timely regeneration turned out to be an important condition because after saturation of the adsorbent, the phosphate concentrations in the drain of the column increase relatively quickly. The adsorption column was regenerated six times, demonstrating that the iron granules can be reused well.

During regeneration, an average of 82% of the captured phosphate removed could be des-orbed again (6 regenerations). The incomplete desorption was most likely caused by insufficient lye consumption during the first regenerations and sometimes by oversaturation of the adsorbent due to late regeneration. On a lab scale, controlled conditions could be used to demonstrate that 90-100% of the phosphate could be desorbed at 10 consecutive cycles (adsorption, regeneration, adsorption, etc.). As the pilot trial progressed and operational stability improved, the desorption rate increased to 88%.

The treatment of the regenerate with nanofiltration showed that 70 -90% of the caustic soda could be recovered with the membrane. The caustic soda is released into the permeate and the phosphate into the concentrate. Lab tests showed 70-90% P-precipitation with calcium from the regenerate.
Treating the regenerate with an NF membrane followed by phosphate recovery using calcium precipitation, therefore seems to be a suitable method for recovery of auxiliary and raw materials for the most sustainable process possible.

BUSINESS CASE

In a business case, the costs of BiOPhree were calculated for a scale of 100,000 PE (625 m^3/hour DWF, current effluent: 0.7 mg P/l on average) for two scenarios:
A: P-requirement of 0.3 mg P/l (annual average)
B: P-requirement of 0.1 mg P/l (annual average)

For scenario A, a BiOPhree installation with a capacity of 750 m^3/hour (1.2 x DWF flow) that treats 70% of the effluent annual flow rate is sufficient. Sce-nario B requires a BiOPhree plant with a capacity of 3,750 m3/hour (6 x DWF flow rate) that treats 100% of the effluent annual flow rate.

The cost-price varies from 0.18 euros/m^3 (scenario A) to 0.53 euros/m^3 (scenario B). For scenario A, the costs were compared with reference techniques such as cloth and sand filtration with coagulant dosing that treat 3 x DWF (90% of the effluent annual flow rate) and thus also arrive at 0.3 mg P/l annual average. The costs of these techniques are in the same range: 0.17-0.22 euros/m^3. For scenario B, there is no reference technique known with practical experience and therefore no comparison could be made.

The CO_2 emissions of BiOPhree (scenario A) amount to about 19 g CO_2/m^3, com-parable to cloth and sand filtration: 18-22 g CO_2/m^3.

BiOPhree is therefore comparable in terms of costs and CO_2 emissions to reference techniques, but has a number of additional advantages:

- In DWF, BiOPhree achieves 0.1 mg P/l where reference techniques are atypically unable to achieve <0.25 mg P/l. Since DWF is treated 75% of the time at a WWTP, this is already a big advantage.
- If BiOPhree is designed for a higher flow rate (> 1.2 x DWF), the P content can drop even further from 0.3 mg P/l to 0.1 mg P/l.
- With BiOPhree, the extra phosphate removed is recovered, with reference techni-
- ques the extra phosphate removed ends up in the sludge with an associated increase in sludge production.

In addition, a number of optimizations will take place for BioPhree due to the no-velty of the technology that are not expected with the reference techniques, which will further reduce the expected costs. BiOPhree is therefore a sustainable phosphate removal and recovery technology that can meet more stringent consent limits than current commercially available reference techniques. For WWTPs that discharge into sensitive surface water (with a strict WFD standard for phosphate), BiOPhree can therefore be an important measure for achieving the WFD targets.

STOWA IN BRIEF

WATER KNOWLEDGE, FOR NOW AND THE FUTURE

The Foundation for Applied Water Management Research – STOWA for short – is the knowledge platform for regional water managers in the Netherlands. Based in Amersfoort, we develop, collect, disseminate, and implement applied knowledge. Water managers need this knowledge to effectively address the challenges they face, whether it concerns climate adaptation (both urban and rural areas), good water quality, sustainable and effective wastewater treatment, safe dikes and quays, energy transition, or the circular economy.

The type of knowledge we develop is broad: technical and scientific, but sometimes also administrative and/or legal. To ensure that this knowledge can be applied in practice, we present our research results, where possible, in the form of reports, practical guidelines, tools, and instruments. We also facilitate learning communities and organize symposia, workshops, and webinars to promote knowledge transfer. To maximize our impact, STOWA explicitly addresses the organizational and human factors that can influence our work. Change is driven by content (knowledge), but it's driven by people. Moreover, we look beyond our own network when seeking solutions. We also look at other sectors, explore different perspectives, and learn from abroad. We explain the background of our work through our own and other media.

STOWA operates demand-driven and also sets agendas. We identify the knowledge questions water managers have and escalate these questions to the appropriate knowledge providers: universities, knowledge institutes, knowledge companies, or consultancy firms. We handle the tendering and supervision of joint knowledge projects. We ensure that water managers remain connected to the projects and have ownership over them. This ensures that the project results offer participants practical action perspectives. Each project is therefore supervised by a committee comprised of regional water managers. The broad research lines are determined by program committees, which include water managers.

STOWA is independent, impartial, and transparent. Users of our knowledge must be able to trust that the content of our reports is objective and representative. Only then can our knowledge be applied to better water management and innovations that address the challenges of today and tomorrow. It is up to water managers to determine how they apply STOWA's knowledge in practice.

STOWA is a foundation that adheres to the guidelines for non-profit organizations (RJ-640). Therefore, our annual report includes both financial statements and a management report on the foundation and its activities. The annual budget is approximately €20 million. Our participants collectively contribute approximately €10 million annually as structural contributions. In addition, we receive approximately €10 million annually in contributions to individual projects.

PILOT STUDY ON ADVANCED PHOPSHORUS REMOVAL FROM WWTP EFFLUENT USING BIOPHREE® TECHNOLOGY

INHOUD

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INTRODUCTION

From October 2023 to December 2024, pilot research into the BiOPhree techno-logy took place at the Dronten WWTP (Zuiderzeeland Water Authority). With this technology, phosphorus can be removed to < 0.1 mg P-tot/l. These very low P levels are interesting for post-treatment of WWTP effluent at locations where strict WFD requirements must be met. Phosphate levels are still too high in many water bodies, leading to eutrophication of surface water (Compendium for the living environment, 2021). At locations where phosphate levels are strongly influenced by the discharge of WWTP effluent, extensive phosphate removal is necessary.

With available techniques such as downstream sand or cloth filtration, phosphate can be removed to approximately 0.25 mg P/l (STOWA 2024 -27), but in some situations an even lower concentration is needed. The BiOPhree technology is then a promising solution.

BiOPhree was originally developed by Aquacare Europe BV (hereafter Aquacare) for treatment of cooling water to prevent biofouling. In principle, biofouling can be reduced by removing extensive phosphate from cooling water. The name is an allusion to this principle; bio free becomes Bio(P)hree.
The technology can also be used for phosphate removal from effluent from a wastewater treatment plant. This has been tested over several years on a lab scale (with artificially contaminated water and with real effluent) and in some shorter pilot tests abroad. This research is the next step in the development of BiOPhree technology. Together with the Zuiderzeeland Water Authority and STOWA, Aquacare and Haskoning have carried out a long-term pilot test with the BiOPhree technology at the Dronten WWTP.

PURPOSE OF THE STUDY

The main objective of the study was to demonstrate a stable phosphate removal from effluent up to approximately 0.1 mg P/l, while maintaining adsorption capacity of the granules in the adsorption column after multiple regenerations. In addition, the recovery of caustic soda and phosphate was investigated because they largely determine the business case and the sustainability of the concept.

COLLABORATION BIOPHREE

Haskoning and Aquacare have been working together since 2020 on the further development of the BiOPhree technology to make it suitable for far-reaching P removal from effluent. In addition, there is collaboration with Wetsus, where fundamental research is being carried out within the phosphate theme into the development of suitable adsorbents for P removal to very low levels. This is outside the scope of this STOWA project.

The pilot study was carried out in collaboration with the Zuiderzeeland Water Authority and STOWA. The water authority has made space available at the Dronten WWTP to be able to carry out the pilot tests. STOWA followed the research results through a supervisory committee and contributed ideas about the translation into a practical installation.

Practical support was provided by HBO[1] students of chemical engineering who did an internship at the Dronten WWTP during the project and by the operators of the Dronten WWTP.

READING GUIDE

This report describes the results of the pilot study and several lab studies on the BiOPhree technology. The report is structured as follows:

- In chapter 2 the BiOPhree technology is explained; Chapter 3 discusses the experimental design; Chapter 4 discusses the results;
- Chapter 5 discusses the application of BiOPhree in practice. The economic feasibility and the most important technical points of attention are discussed;
- Chapter 6 concludes with conclusions and recommendations.

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[1] University of applied sciences

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THE BIOPHREE® TECHNOLOGY

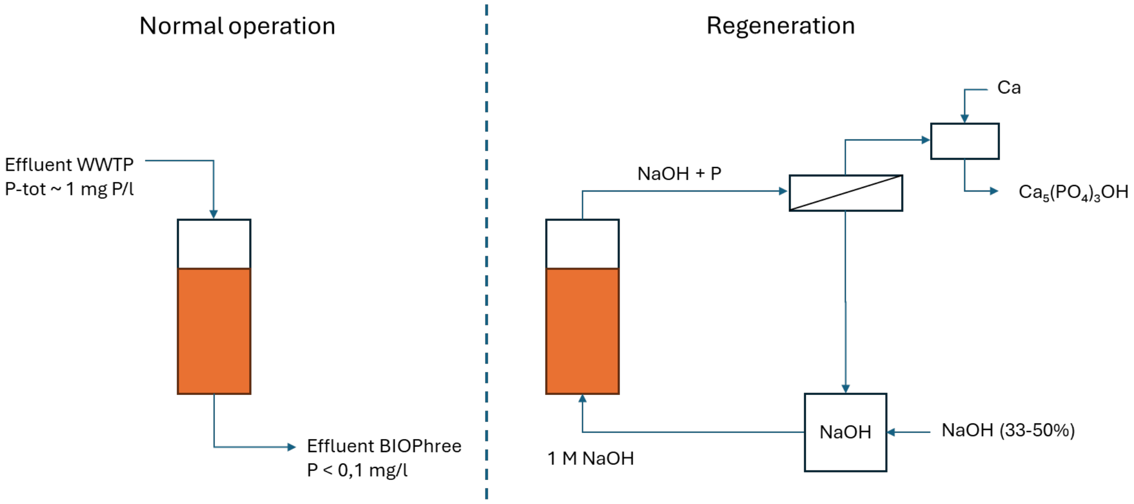
The BiOPhree technology is based on phosphate adsorption to iron oxyhydroxide (FeOOH) granules (hereafter abbreviated to iron oxides or iron granules). These granules are produced by various suppliers, mainly for arsenic removal. Arsenic is present in groundwater as arsenate ($AsO4^{3-}$) and is well bound to iron oxides by the negative charge.
Phosphate (PO_4^{3-}) is very similar to arsenate and also wants to bind well to iron granules. Various studies have already shown that the phosphate releases (des-orbs) at a high pH (see e.g. Sperlich et al, 2010). The iron granules can therefore also be used for phosphate removal instead of arsenic removal.

The BiOPhree process basically works as follows:

1. WWTP effluent is filtered to remove suspended solids.
2. The effluent is then passed over a solid bed of iron granules (the BiOPhree column) where the dissolved phosphate is bound by adsorption.
3. The outgoing water has a P content of < 0.1 mg P/l until the column is saturated and a break-through of phosphate occurs. The P levels then start to rise towards 0.2 -0.3 mg P/l.
4. After saturation of the adsorbent, regeneration is required. The bound phosphate is desorbed by rinsing the column with caustic soda (NaOH). The adsorbent is then suitable for the next adsorption cycle.
5. After regeneration, the caustic soda can be treated with a nanofiltration membrane in which the majority of the caustic soda is recovered from the permeate and can thus be reused for subsequent regeneration.
6. Phosphate is largely retained by the membrane and can be recovered from the concentrate by precipitation with calcium or another cation.

Figure 1 shows the different steps of BiOPhree schematically.

Figure 1 **Schematic representation of the BiOPhree process**



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PILOT DESIGN

3.1 PILOT RESEARCH

3.1.1 LOCATION

The pilot study with the BiOPhree technology took place at the Dronten WWTP of the Zuiderzeeland Water Authority. This treatment plant has a biological capacity of 65,000 P.E.$_{150}$ and consists of a conventional activated sludge system with pre-settling. Phosphate and nitrogen are biologically removed (m-UCT configuration) with additional simultaneous phosphate precipitation with poly-aluminum chloride (PAC). Primary and secondary sludge are digested at the WWTP and then dewatered with belt presses.

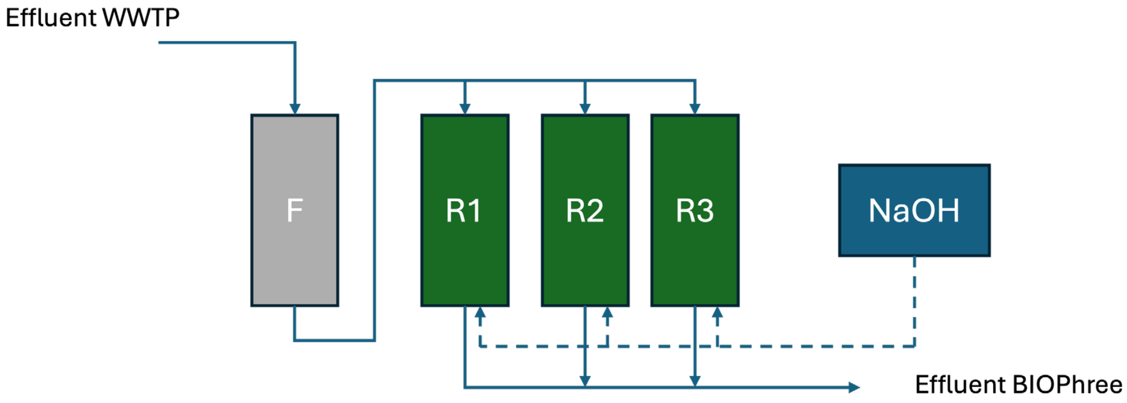
The pilot plant was fed with the effluent from the WWTP. During the test period, this effluent had an average phosphorus concentration of 0.91 mg P_{tot}/l and 0.83 mg PO_4-P/l. The concentrations varied greatly from day to day, see chapter 4. Chapter 3.5.1 also shows the concentrations of other parameters for the effluent from the Dronten WWTP and for the effluent from the Den Bosch WWTP that was used for lab tests.

Figure 2 **Photo of WWTP Dronten, Zuiderzeeland Water Authority**

3.1.2 PILOT INSTALLATION

The pilot installation consisted of a prefiltration and three parallel adsorption columns (Figure 3 and Figure 4).

Figure 3 Simplified process diagram BiOPhree pilot. F: filter, R1 to R3: BiOPhree adsorption columns, NaOH: NaOH storagetank



TREATMENT OF THE EFFLUENT

The effluent from the WWTP was first treated with a multimedia filter to remove suspended solids. The filter bed in this type of filter is similar to a sand filter. The filter was backwashed with BiOPhree effluent several times a day to wash out accumulated suspended solids. The backwash water was discharged into the sewer. After the pre-filtration, the water was passed over the BiOPhree adsorption columns (from top to bottom) for extensive P removal. Typically, two of the three identical columns were fed and operated in parallel. The third column was on standby. In the last month of the test, only column 1 was used to achieve a higher hydraulic loading rate. After the adsorption columns have passed, the BiOPhree effluent was discharged via a buffer tank into the WWTP's site sewer system.

Figure 4 Left: Inside of the pilot (in the foreground the multimedia filter, behind it three adsorption columns and NaOH tank) Right: Container in which the pilot was placed at the Dronten WWTP

ADSORBENT AND CONTACT TIME

The adsorption columns were filled with iron granules that allow phosphate to be selectively removed from the effluent by means of adsorption. During the pilot tests, two types of granules were tested:

Table 1 **Iron granules tested in the pilot study**

Iron granules	Grain size	Bulk density	Surface Spec
	Mm	kg/m^3	m^2/g
GEH	0,2 – 2,0	1150	300
GFH	0,3 – 2,0	530	150

Both types of iron granules therefore differ in terms of specific surface area and bulk density. The density of Granular Eisen Hydroxide (GEH) is based on wet granules, the density of Granular Ferric Hydroxide (GFH) is based on dried granules. Under equal conditions, the density of both grains is almost the same.

The contact time of the water with the adsorption bed is usually expressed as Empty Bed Contact Time (Empty Bed Contact Time; EBCT) This is calculated by dividing the total bed volume by the flow rate. The EBCT on which the adsorption columns were operated varied from 5 — 10 minutes. The volume of the adsorption bed was about 160 liters.

REGENERATION OF THE ADSORBENT

Over time (after approximately 3,000 bed volumes[2]), the adsorbent became satu-rated with phosphate and phosphate breakthrough occurred. The phosphate concentration in the effluent of the adsorption column rose to values above 0.1 mg P/l. From this moment on, regeneration had to be done with a caustic soda solution. Two regeneration strategies were applied in the pilot study; stagnant regeneration and single-pass regeneration.

Stagnant regeneration

In the first phase of the project (Oct 2023 to January 2024), a limited amount of NaOH (1 M) (approximately 0.7 bed volumes) was used during regeneration by filling the adsorption column from bottom to top with NaOH, waiting approximately 30 minutes and then emptying it.

Single-pass regeneration

When stagnant regeneration proved to be insufficient, the pilot was adapted so that single-pass regeneration could be applied. During this regeneration, the column was regenerated with approximately 5 bed volumes caustic soda. Caustic soda was pumped through the column from bottom to top and collected at the top. The column was rinsed with softened water before and after regeneration to lower the pH and prevent calcium precipitation. This strategy has been tested from May 2024 to December 2024.

3.1.3 PHASING OF THE PROJECT

The pilot test is split into two phases (see Figure 5 and Table 2). In phase 1 (Octo-ber 2023 to January 2024), stagnant regeneration was applied. Because this proved insufficiently successful, a number of adjustments were made to the pilot so that the improved regeneration strategy (single pass regeneration) could be applied in phase 2 (May to December 2024). In phase 1, columns R2 and R3 were in use. In phase 2, columns 1 and 2 were in use. In phase 2, new iron granules were also started, with column 1 filled with GFH granules and column 2 with GEH granules. Between phase 1 and phase 2 (January-May 2024) the pilot did run but without regenerating the adsorbent.

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[2] A bed volume is equal to the total volume occupied by the iron granules in a column.

Figure 5 **Phasing of the Pilot Project BiOPhree, in phase 1 stagnant regeneration was applied, in phase 2 single pass regeneration**

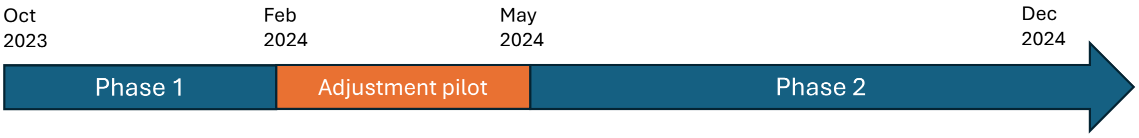

Table 2 **Phase 1 and phase 2 pilot installation settings**

	Unit	Phase 1	Phase 2
Columns in operation	n	R2 and R3	R1 and R2
EBCT	minus	10	5-10
Type of regeneration		Stagnant	Single pass
Correlate type		GEH	R1 (GFH) R2 (GEH)
Operating hours	hours/day	24	24

3.1.4 SAMPLING AND ANALYSES

PHOSPHATE REMOVAL

The pilot was sampled on a daily basis. A 24-hour flow proportional sample was taken from the effluent from the WWTP (supply of the pilot) 7 days a week. A grab sample was taken from the effluent of the BiOPhree columns from Monday to Friday. The samples were analyzed for Total Phosphorus (TP), PO_4-P and pH.

REMOVAL OF OTHER COMPONENTS

At the end of August – beginning of September, a 24-hour flow proportional sample of the effluent from the WWTP and an effluent sample of column 1 and column 2 were taken on 8 different days and analyzed with ICP-OES[3] (iCAP PRO, Thermo Scienti-fic) for metals and P, K and S. In addition, COD was incidentally measured with Hach Lange cuvette tests.

REGENERATIONS

During the various regenerations, samples were taken of the regeneration solution (the caustic soda solution that is released during regeneration of the column) to determine the amount of P desorption that occurred during regeneration. These samples were also analyzed with ICP-OES.

With cuvette test LCK514, COD was also determined in the regeneration samples. The DOC (dissolved organic carbon) and humic acids of a few regeneration samples were also determined by an external laboratory (DOC-La-bor, Mannheim).

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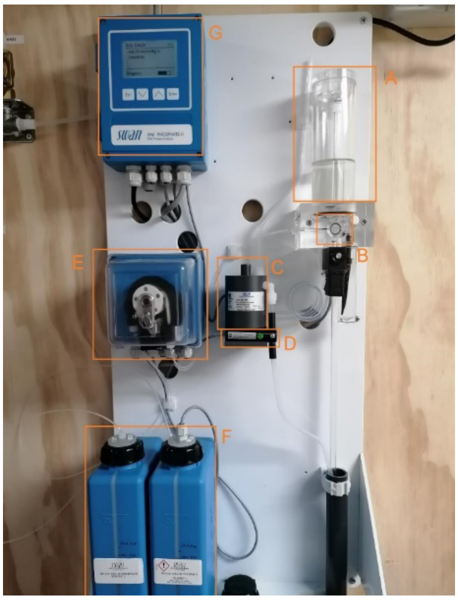
[3] Inductively coupled plasma optical emission spectroscopy

Figure 6 **Online PO_4-P monitor**



ONLINE PHOSPHATE MONITOR

The pilot was equipped with an online phosphate monitor (AMI-Phosphate II, Swan Analytical instruments) (Figure 6). This was used to alternately measure or-thophosphate in the supply of the pilot (after pre-filtration) and in the outcome of the three columns. The measurement range of the analyzer was 0.1 – 16 mg PO_4-P/l according to the specifications. Prior to the study, it was not clear what the measurement accuracy was in the low measurement range (around 0.1 mg P/l), which is why the analyzer was only used in this project to investigate whether it is suitable for detecting phosphate breakthrough.

OTHER ONLINE MEASUREMENTS

Furthermore, the pilot was equipped with the following online measurements:

- flow measurement per column;
- pressure measurements per column (both in the supply and in the discharge);
- pH measurement in the joint discharge pipe of 3 columns.

Table 3 **Offline and online analyses and measurement frequency supply and outcome of pilot and regeneration**

Parameter	Method of analysis	Frequency
P-tot	Hach Length LCK349	Daily
PO_4-P	Hach Length LCK349	Daily
PO_4-P	Online analyser	online
COD	Hach Length LCK314	Incidental
pH	pH Meter	Daily
Elements	ICP-OES	Incidental

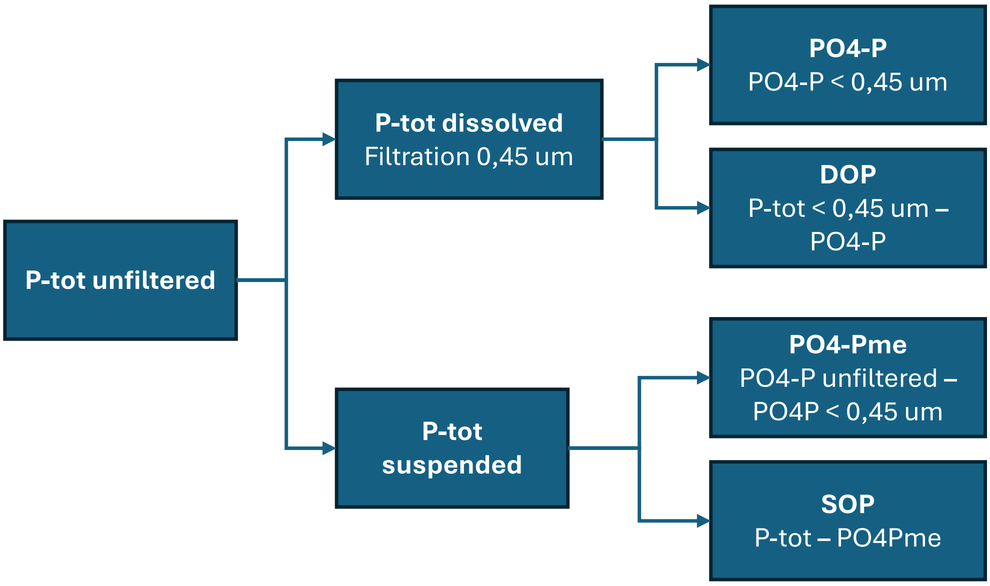
[1] For several weeks, before and after the pilot, P-to and PO 4-P were determined in unfiltered and filtered samples (with a 0.45 µm filter) in order to be able to derive the P-fractions.

3.2 CALCULATING P FRACTIONS

During the pilot trial, the P fractions were incidentally determined in the supply to the pilot and the effluent of the columns. The P-fractions were determined by measuring P-tot and PO_4-P twice in the samples: before and after filtration of the sample over a 0.45 µm filter.

This was not done for all samples, but with a frequency of about once a week in period A and about once every two days in period B. The calculation of the P fractions from the P analyses is shown below:

Figure 7 **P-fractionation effluent WWTPs (SOP: suspended organic phosphorus, DOP: dissolved organic phosphorus, PO_4Pme: metal-bound ortho-phosphate, P-tot; total phosphate, PO_4-P: ortho-phosphate)**



3.3 MASS BALANCE CALCULATIONS

In order to properly evaluate the adsorption and desorption of phosphate, a mass balance has been established across the column:

1. P-adsorption = Feed flow rate * ($\text{P-}_{\text{tot, feed}} - \text{P-}_{\text{tot, effll. column}}$)
2. $\text{P-}_{\text{adsorption spec.}}$ = $\text{P-}_{\text{adsorption}}$ / granules in column
3. $\text{P-}_{\text{desorption}}$ = $\text{P-}_{\text{tot,regenerated}}$ * $\text{Volume}_{\text{ Regenerate}}$ + $\text{P-}_{\text{tot, Rinse Water}}$ * $\text{volume}_{\text{ flushwater}}$
4. $\text{P-}_{\text{desorption spec.}}$ = $\text{P-}_{\text{desorption}}$/granules column"

Wherein:

- $\text{P-}_{\text{adsorption}}$: Adsorbed P of iron granules per cycle (g P);
- $\text{P-}_{\text{adsorption spec}}$: Specific P-adsorption per kg Fe-granules (g P/kg);
- $\text{P-}_{\text{desorption}}$: Desorbed P per regeneration (g P);
- $\text{P-}_{\text{desorption spec}}$: Desorbed P per kg Fe-granules (g P/kg);
- Feed flow rate : Effluent flow rate to pilot (m^3/hour)
- $\text{P-}_{\text{tot, feed}}$, $\text{P-}_{\text{tot, end column}}$: P-Levels Closed and Discharge Pilot (g P/m^3)
- $\text{P-}_{\text{tot, regenerate,}}$ $\text{P-}_{\text{tot,rinse water}}$: P-contents of regeneration and rinse water regeneration (g P/m^3);
- Granules column : Quantity of granules in 1 column (kg)
- $\text{Volume}_{\text{ regeneration}}$: Amount of NaOH per regeneration (m^3)
- $\text{Volume}_{\text{ flush water}}$: Amount of flush water per regeneration (m^3)

Note: because no sample was taken of the effluent of the BiOPhree column during the weekends, the following was assumed for a mass balance:

- P-effluent BiOPhree on Saturday is the same as P-effluent BiOPhree on Friday.
- P-effluent BiOPhree on Sunday is the same as P-effluent BiOPhree on Monday.

Since the phosphate concentration in the effluent of the BiOPhree columns[4] is con-sistently quite low (< 0.2 mg P/l P/l) until the moment of breakthrough (when > 0.2 mg/l P is reached), the above method is quite accurate and suitable to get a good indication of the P removal in the weekend.

3.4 MEM BRANETEST NAOH - RECOVERY

Recovery of caustic soda from the regeneration liquid has been tested using nano-filtration in a pilot set-up (see Figure 9) at EMI-Twente in Enschede (a research institute affiliated with the University of Twente that conducts contract research in the field of membrane technology). Various membrane tests were carried out with a spiral-

wound NF module of 2.5'' (membrane area 1.6 m^2) at a flow rate of 80 -200 lit-res/hour and a pressure of 35 bar. Figure 8 shows the principle of membrane fil-tration. Approximately 500 litres of regeneration liquid from one regeneration were used for the membrane test.

Figure 8 **Principle of recovery of NaOH with nanofiltration**

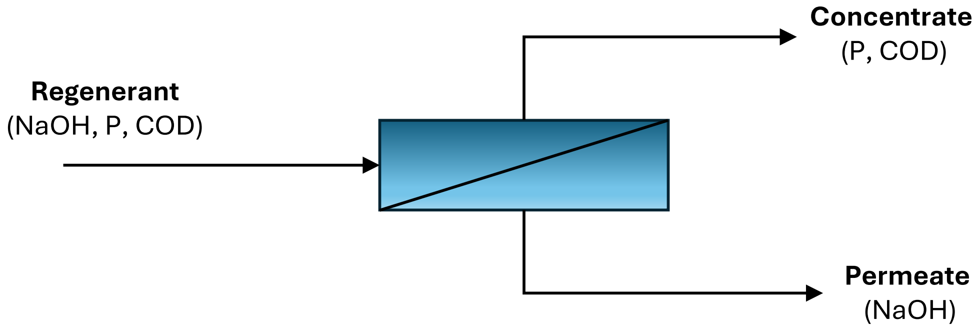
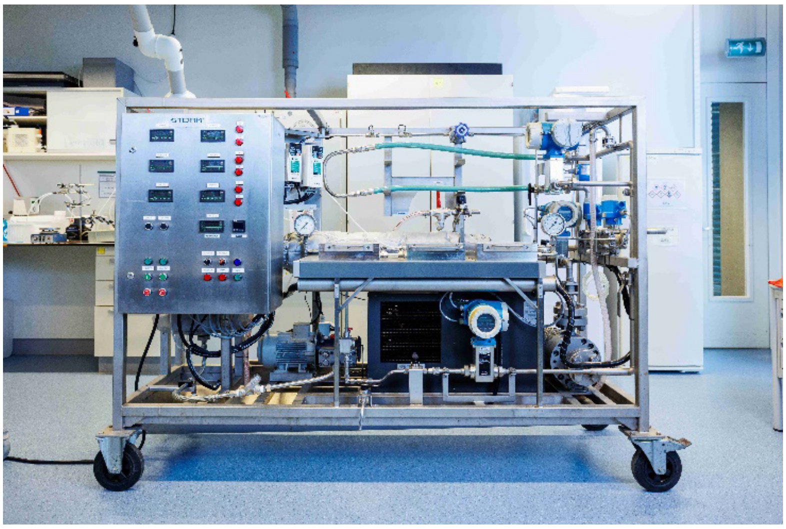


Figure 9 **Photo of the nanofiltration test set-up at EMI-Twente**



The following parameters were monitored during the tests:

- Pressure drop across the membrane (bar);
- Flux through the membrane and course over time (l/m^2.hr);
- Recovery (proportion of permeate to feed) (%);
- Retention of phosphate, COD, NaOH and conductivity (%).

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[4] In the pilot test, for practical reasons, a cycle could not always be stopped if the P content was > 0.1 mg P/l. The P content at the end of a cycle averaged about 0.2 mg P/l.

3.5 ADDITIONAL LAB RESEARCH

Several additional lab tests have been carried out at Aquacare and Haskoning. These are explained in this section for each topic.

3.5.1 COLUMN TESTS AQUACARE

Aquacare has tested the BiOPhree technology on a lab scale with small columns. The aim was to evaluate the improved regeneration strategy, which was tested in phase 2 in the pilot, under more controlled conditions at lab scale. During these tests, 2 adsorption columns of 50 ml, filled with GFH granules, were used. The columns were fed with effluent from the Den Bosch WWTP to which phosphate had been added up to 4 mg P/l. This relatively high concentration was used so that the columns became saturated faster, and more regeneration cycles could be carried out. In total, the columns have been regenerated ten times. Table 4 shows the composition of the effluent from the Den Bosch WWTP and the Dronten WWTP (average of August and September 2024).

Table 4 **Composition of effluent at the Den Bosch and Dronten WWTPs**

Parameter	Unit	WWTP Den Bosch[1]	WWTP Dronten[2]
P	mg/l	2.9 ± 1.0	0.93 ± 0.7
S	mg/l	9.3 ± 2.4	17.2 ± 1.8
Ca	mg/l	31.2 ± 7.7	52.9 ± 5.3
Si	mg/l	6.3 ± 1.7	6.9 ± 0.8
Mg	mg/l	5.8 ± 1.0	8.1 ± 1.1
pH	-	7.4 ± 1.8	7.7 ± 0.3

[1] average of 25 days

[2] average of 29 days, except P and pH (average over the pilot period)

The conditions under which the columns were operated for phosphate adsorption and regeneration were as follows:

- Pre-filtration over (from top to bottom): anthracite (0.8-1.6 mm), sand (0.5-1 mm) and gravel (1-2 mm);
- Empty bed contact time: 5 minutes;

3.5.2 CALCIUM PRECIPITATION TESTS

With the spent regenerant produced during the regeneration of the adsorption columns on the pilot installation, and with the concentrate produced during the membrane tests, a number of calcium precipitation tests were carried out to gain insight into phosphate removal at different molar ratios of Ca/P.

The tests were carried out with 250 ml beakers at room temperature. The procedure was as follows:

1. Concentrate or spent regenerant was added to 250 ml beakers;
2. Next, $CaCl_2$ solution was added (0.8 M) while the solution was mixed with a magnetic stirrer (about 100 rpm);
3. After a reaction time of 5 minutes, the stirring stopped;
4. The solution was allowed to settle for 10 minutes;
5. A sample was taken from the above water layer and analyzed for phosphate using a Hach Lange cuvette test (LCK314).

3.5.3 SUSPENDED SOLIDS REMOVAL IN BIOPHREE COLUMN

The effluent from the Dronten WWTP was filtered with a multimedia filter to re-move suspended solids before it was pumped into the BiOPhree column. This is to prevent clogging of the column. However, on a practical scale, this pre-filtration is still quite expensive and takes up a relatively large space. Therefore, an initial study was carried out on lab scale to test whether the filtration step can be omitted and the adsorption column itself can capture the suspended solids.

Removal of suspended solids in the adsorption column requires regular backwashing to flush out the captured particles. Two questions were addressed in the lab tests. Firstly, how well the suspended solids can be separated from the iron granules and what backwash speed is needed to flush out suspended solids whilst keeping the granules in the column. Second, it was also investigated what the effect of backwash is on the P stratification[5] in the column. By regularly backwashing the column, the P-stratification is likely to be disrupted since the iron granules are suspended and mixed every time there is a backwash, which may lead to a faster breakthrough of phosphate.

In these exploratory lab scale experiments, , the upflow velocity required to separate suspended solids from iron granules and the effect of backwashing on the P-stratification of the adsorbent were investigated.

SEPARATION OF SUSPENDED SOLIDS AND GRANULES

A column with a diameter of 5 cm was filled up to a third of its height with GFH granules and fed with effluent from the Amersfoort WWTP to which 50 mg/l suspended solids (thickened surplus sludge) had been added. The column was then backwashed at different speeds (5-20 m/hour) and samples were taken from the effluent for determining suspended solids concentration. Losses of iron granules were determined visually..

BACKWASH EFFECT ON STRATIFICATION

A lab column with a diameter of 2 cm was filled up to a third of its height with GFH granules and loaded with phosphate by feeding it with effluent spiked to 40 mg P/l. The column was then backwashed once at a rate of 20 m/hour for 5 minutes. After settling of the bed, samples were taken from different heights in the bed to gain insight into the P-stratification. The samples were analyzed with ICP-OES by Aquacare. The test was repeated with a column that was not backwashed so that the measurement result could be compared with a reference.

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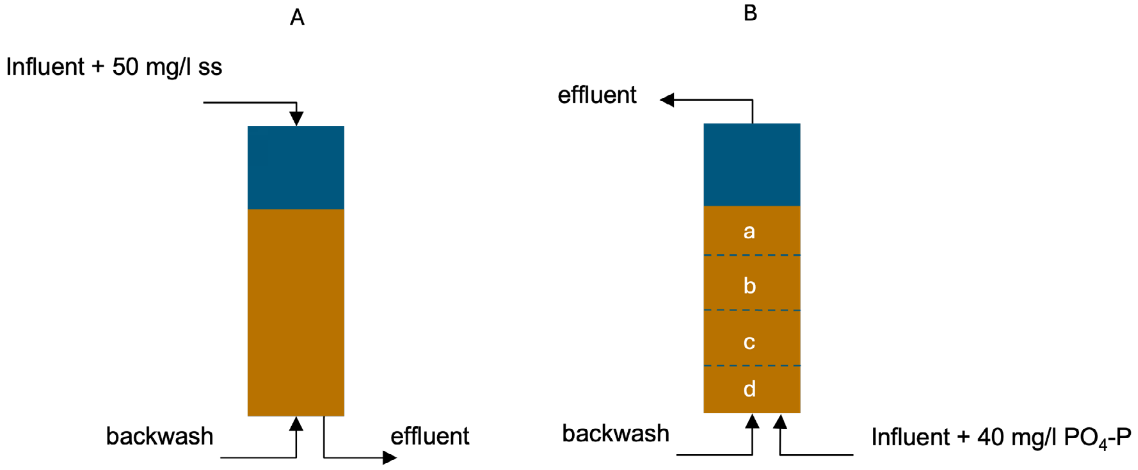
[5] An adsorption column gradually saturates from top to bottom and will contain the highest P levels at the top and the lowest at the bottom.

Figure 10 Scheme lab scale experiment A: suspended solids capture with backwash, B: P-loading with backwash



4

RESEARCH RESULTS AND DISCUSSION

In chapters 4.1 to 4.3, the results of the BiOPhree pilot tests, the membrane tests and the lab tests are presented.

4.1 PILOT TEST

4.1.1 P REMOVAL PHASE 1

The pilot started on 4th October 2023 with columns 2 and 3 being filled with GEH granules and operated until January 2024. The P concentration in the influent and effluent of column 2 are shown in Figure 11 and of column 3 in Figure 12. The influent P concentrations fluctuate around 1 mg P/l. The effluent P concentrations remained < 0.1 mg P/l in the first weeks (until mid-November). In this period, almost 90% of the phosphorus in the effluent was removed. From mid-November onwards, breakthrough took place, with P concentrations in the effluent > 0.1 mg P/l, as shown in Figure 11 and Figure 12. Both columns were then regenerated using a procedure referred to as stagnant regeneration, see chapter 3.1. Column 3 was regenerated at the end of November and column 2 was regenerated in mid-December. The figure shows a short decrease in the P effluent concentration after both regenerations, but in both columns the P levels then rises again fairly quickly to > 0.2 mg P/l. In mid-December and late December, both columns were regenerated again, which again resulted in a short decrease in the P effluent concentration. After a short stop due to frost in early January, the P effluent concentration in both columns rises to 0.3 – 0.5 mg P/l. The stagnant regeneration therefore had insufficient effect, and more intensive regeneration was necessary for reaching a higher degree of P desorption. The pilot was adapted for this purpose and the columns were refilled with iron granules. The next section presents the results after these changes.

Figure 11 TP concentration in the pilot influent and the effluent of column 2 in phase 1 (GEH-granules). The vertical gray dotted lines indicate the regeneration cycles. During this period, the concentration in the pilot influent exceeded 2 mg P/l three times. These data points are not displayed

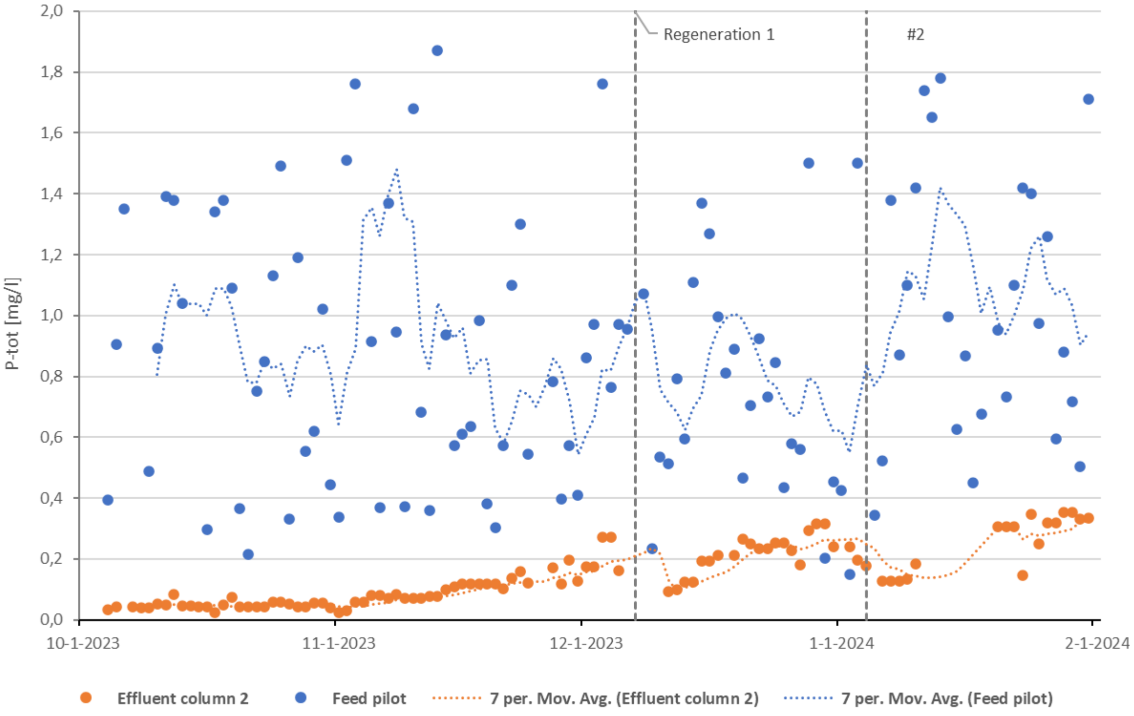
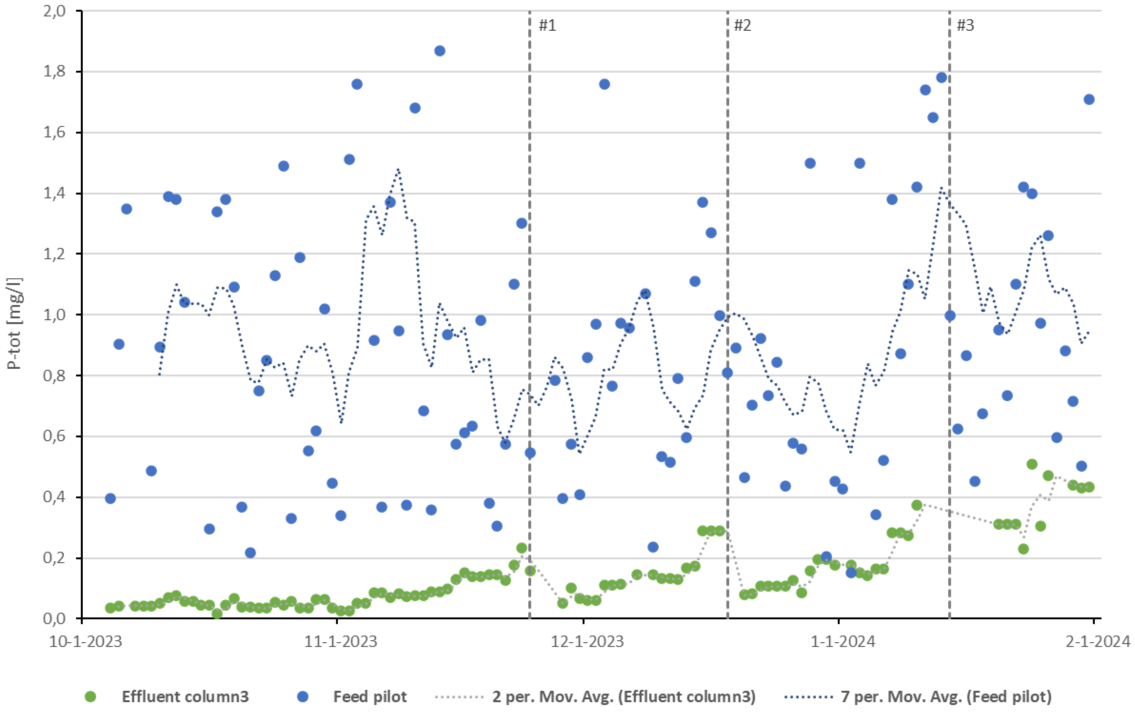


Figure 12 TP concentration in the pilot influent and the effluent of column 3 in phase 1 (GEH granules). The vertical gray dotted lines indicate the regeneration cycles. During this period, the concentration in the pilot influent exceeded 2 mg P/l three times. These data points are not displayed



4.1.2 P REMOVAL PHASE 2

In this phase, an improved regeneration strategy was used, as described in chapter 3.1. Column 1 was filled with GFH granules and column 2 with new GEH granules. The granules from phase 1 were removed to avoid influence of incomplete regeneration (P accumulation in the column) on the results. Figure 13 and Figure 14 show the P removal for both columns from May to December 2024.

Figure 13 TP concentration in the pilot influent and effluent of column 1 in phase 2 (GFH granules). The vertical gray dotted lines indicate the regeneration cycles (1 to 6)

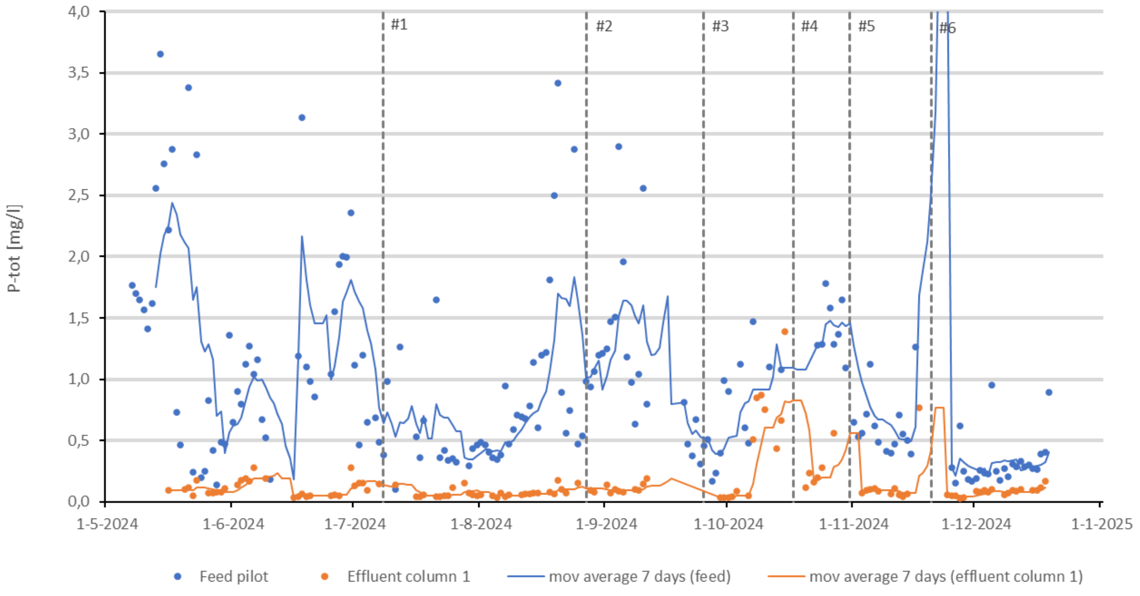


Figure 14 TP concentration in the pilot influent and the effluent of column 2 in phase 2 (GEH granules). The vertical gray dotted lines indicate the regeneration cycles (1 to 5)

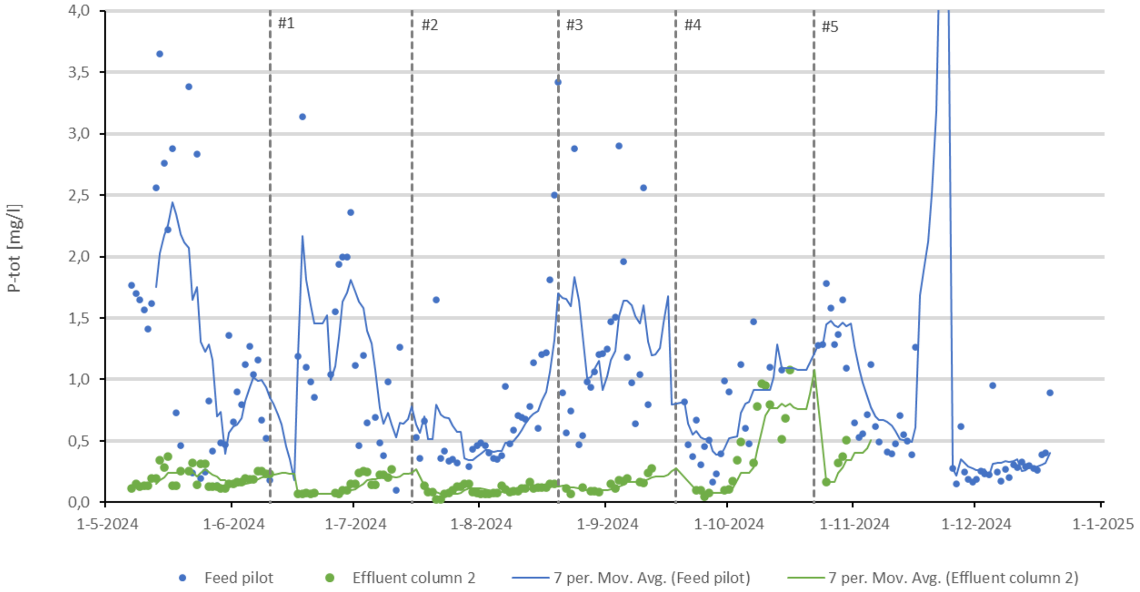


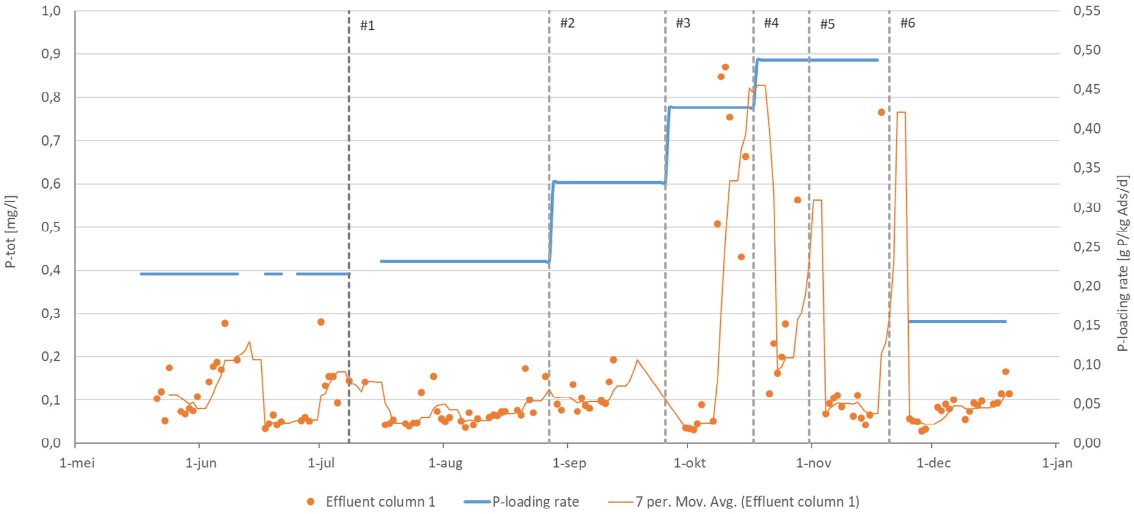
Figure 13 and Figure 14 show that, despite fluctuating P levels in the influent of the pilot, the P level in the effluent remained mostly low (< 0.1 mg P/l). Figure 15 shows the TP concentration in the effluent of column 1. The average P-load of the column per cycles is shown, instead of the P concentration in the influent. This figure shows the effect of the different regenerations. The following points are highlighted:

- In the first cycle, breakthrough seems to happen quickly, with P levels in the effluent up to almost 0.3 mg P/l at the end of the cycle. The fairly rapid breakthrough can partly be explained by fairly high P levels in the influent (2 -3 mg P/l on some days).
- Regeneration 1 shows a clear effect, with decrease in the P concentration in the effluent to < 0.1 mg P/l. The P level then temporarily rises to > 0.1 mg P/l and after that remains < 0.1 mg P/l for a long period.
- Regeneration 2 leads to P-levels that fluctuate around 0.1 mg P/l for several weeks, higher than after regeneration 1. The limited effect of regeneration 2 is related to incomplete P desorption due to the low amount of caustic soda used in the regeneration, see also chapter 4.1.3.
- Regeneration 3 leads to P levels < 0.1 mg P/l followed by a breakthrough to 1.4 mg P/l. During this period, the column was heavily loaded by high P levels in the influent. The P removal at the WWTP was not stable throughout the year, see the blue line in Figure 15 (period October).
- Regeneration 4 to 6 show a decrease in the P content and then an increase again due to saturation of the adsorbent. In the last cycle (after regeneration 6), the P levels in the effluent remain < 0.1 mg P/l for a long time.
- The relatively rapid saturation of the adsorbent after regeneration 4 is due to relatively high P levels in the influent of the pilot and thus a higher P loading of the column.

The P loading of the column is determined by the P concentration in the influent of the pilot (effluent from the WWTP) and the flow rate over a column. Since the hydraulic load of the columns has been increased in the course of phase 2 and thus the EBCT has been reduced, the change in load of the columns is not clearly visible in the figure. This is discussed in more detail in section 4.1.3.

In general, it can be said that the regeneration in this phase worked more effectively for both columns than in phase 1, when the P concentrations in the effluent of the columns slowly increased. In phase 2, it was possible to reach P effluent concentrations < 0.1 mg P/l after 6 regenerations.

Figure 15 **TP in effluent of column 1 (with moving average for the last 7 days) and average P load column 1. The vertical gray dotted lines indicate the regeneration cycles (1 to 6)**



Columns 1 and 2 were regenerated a total of six times in phase 2. It was noticeable that the breakthrough in column 2 (filled with GEH granules), after regeneration was much faster than in column 1. These results were in line with results of lab tests of Aquacare in which both types of granules were tested. A stable adsorption capacity of a column after a regeneration is essential for long-term operations. Based on the lab and pilot tests realized so far, GFH seems to be the best iron granule for a practical application, which is why the P-adsorption and desorption exclusively in column 1 are discussed here.

4.1.3 P- ADSORPTION AND DESORPTION COLUMN 1

Table 5 shows the P adsorption and desorption of the 6 regeneration cycles of column 1 in phase 2. In the last cycle, the column was loaded but no longer regenerated, because the pilot was discontinued.

For each cycle, the following is shown in the table:
- average TP concentration in the effluent of the column;
- adsorbent load (g P/kg adsorbent);
- P desorption as a % of adsorbed P;
- throughput in bed volumes (BV) of WWTP effluent treated up to regeneration;
- the empty bed contact time (EBCT) applied;
- the NaOH consumption during regeneration, in bed volumes (BVs).

Table 5 **The adsorption and desorption of P in column 1 for 7 regeneration cycles**

Cycle	Period	P-tot effluent concentratoin mg/L	Adosrbent load g/kg	Desorbed P %	Throughput BV	EBCT min	NaOH BVs
1	17/05-08/07	0,12	8,2	30%	4506	11,9	3,7
2	08/07-27/08	0,07	8,8	33%	5605	7,9	3,7
3	27/08-25/09	0,09	5,8	64%	3080	9,0	5,8
4	25/09-17/10	0,37	5,2	87%	2784	6,7	5,4
5	17/10-31/10	0,21	5,3	106%	1773	6,4	4,6
6	31/10-20/11	0,12	5,6	71%	2370	5,9	10,0
7	20/11-20/12	0,08	2,9	-	6352	5,0	-

The table is explained in more detail below.

AVERAGE P CONCENTRATION

The average P concentration in the effluent of the column is quite low in the first three cycles (about 0.1 mg P/l) and significantly higher in the fourth and fifth cycles. In the last two cycles, the P concentration is comparable to the first three cycles. The reason for the increased P effluent concentration in the fourth and fifth cycles is probably the incomplete desorption during the previous regeneration. As a result, too much P accumulates and the adsorbent becomes saturated more quickly. The moment of regeneration is also important. For practical reasons, this could only take place a week after the breakthrough had been observed, causing P to continue to accumulate on the adsorbent. In the last week, after saturation of the adsorbent, the P levels sometimes increase rapidly, which affects the average P content in a cycle.

P ADSORPTION

The P adsorption on the granules is high at first (around 8 g P/kg granules), and stabilizes from the third cycle at around 5 – 6 g P/kg. A high adsorption capacity for the first and/or second regeneration was seen more often in lab column tests previously done by Aquacare and is probably related to an equilibrium that has yet to be established. Part of the adsorbed P may penetrate deeper into the granules and cannot be desorbed. Once this has happened, the P adsorption stabilizes at a lower level and remains relatively constant.

In the last cycle, there is a significantly lower adsorption compared to the six previous cycles. It should be noted that during this period there were low P levels in the influent of the column (on average 0.32 mg P/l), which meant that only after about 6,000 bed volumes (just before Christmas) a breakthrough occurred. The explanation for this is that the maximum P adsorption depends on the P content in the influent of the column because there is an equilibrium between the P content in the water and the P content on the granules. The adsorption capacity increases with a higher P content in the water phase. In addition, sulphate and COD are also removed (see chapter 4.1.7) and there is probably more competition for adsorption sites if there is proportionally more sulphate and COD present in the water at low P levels.

P DESORPTION AND CAUSTIC SODA CONSUMPTION

The P desorption is very limited, especially in the first two cycles, at about 30%, so that a relatively large amount of P accumulates in the column. From the third cycle onwards, a much higher P desorption is achieved of an average of 82% in cycles 3 to 6. There are two reasons for this: the higher NaOH consumption (on average 5 bed volumes in cycles 3 to 5 and against 10 bed volumes in cycle 6 alone) and the better neutralization after regeneration. By rinsing the column with softened water, the column was neutralized after regeneration to pH 8 from cycle 2 onwards. As a result, less calcium precipitation could take place and a higher P desorption was achieved. It is important to note that regeneration with 10 bed volumes caustic soda in a single cycle (regeneration cycle 6) does not seem to have had a positive effect on the P desorption. It is not clear why this was the case.

BED VOLUMES TREATED AND EBCT

The number of bed volumes that were treated up to a regeneration varied from ap-proximately 1,800 to more than 6,000. The P loading of the column (P content in supply x flow rate) mainly determines the number of bed volumes up to breakthrough. The average P load per cycle ranged from about 13 g P/d to 41 g P/d (see Figure 15). The figure shows that with a higher P load, regeneration is more frequent.

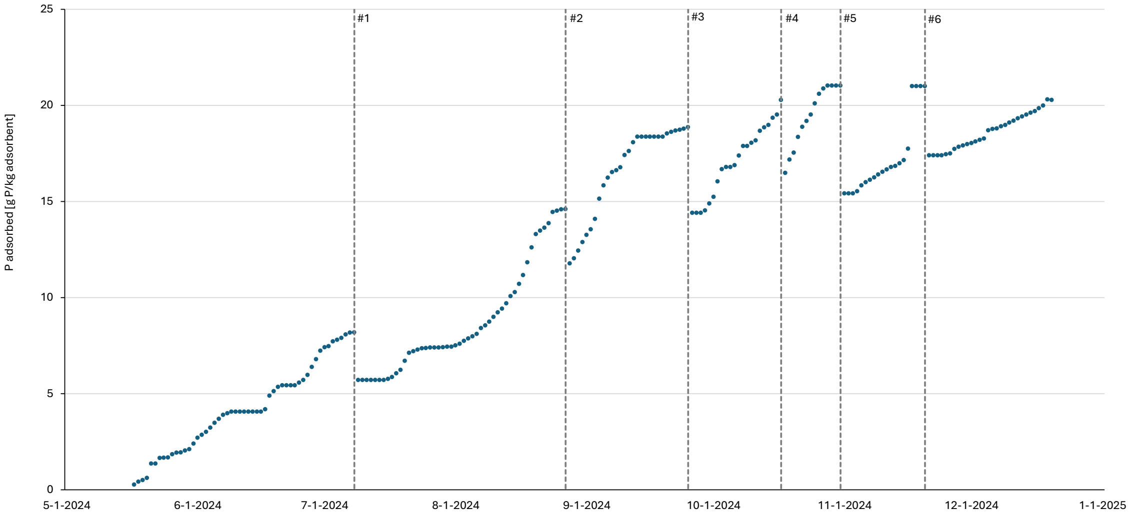
During phase 2, the empty bed contact time (EBCT) has been further reduced because this parameter is important for the design of a full-scale plant (the lower the contact time, the more compact and less costly the reactor is). The results showed that an EBCT of 5-6 minutes still resulted in low P levels in the effluent.

Figure 16 shows the P adsorption and desorption of column 1 for the period from 17 May to 20 December 2024. The P accumulation (also shown in the incomplete desorption, Table 5) is clearly visible here. Over time, the amount of P in the column increases steadily. From the 4th cycle onwards, P adsorption and desorption are more balanced and the operation is more stable.

It is important to note that in the sixth cycle, there is a very strong increase in P adsorption towards the end of the cycle: about 200 g of P increase in 1 day. The reason for this is a very high P content in the influent of the pilot (8 mg P/l). this was a day of wet weather flow at the WWTP when there was also resulting in poor P removal at the WWTP. did not function properly. It can be confirmed that this is not a measurement error since the online measurements in the activated sludge also showed similar values.

Whether the pilot was also constantly fed with 8 mg P/l on this day is not certain.
The effluent from the WWTP was sampled flow proportionally over 24 hr and the measured 8 mg P/l is the result of the average concentration in the effluent on an rain weather flow day. However, the pilot was fed at a constant flow rate and probably had a lower average P concentration in the influent than the P concentration in the flow rate proportional effluent sample. As a result, the total adsorption in this cycle is likely to be lower and the percentage P desorption is slightly higher than the values for cycle 6 in Table 5.

Figure 16 **P adsorption and desorption for column 1 (GFH) in phase 2. The vertical gray lines indicate the regeneration cycles**

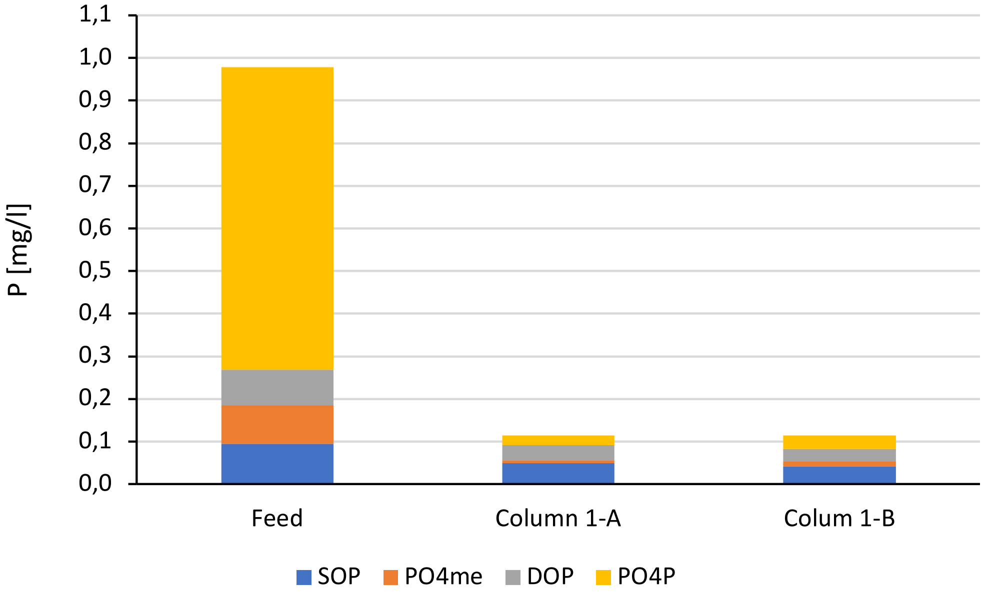


4.1.4 P FRACTIONS BEFORE AND AFTER COLUMN

Figure 17 shows the average P fractions in the influent of the pilot in two periods (A and B) and the effluent of column 1 for both periods separately. The figure shows that in both periods the TP concentration decreases sharply over the column to 0.11 and 0.12 mg P/l respectively in periods A and B. Furthermore, it appears that the decrease is mainly caused by PO$_4$-P removal, but that the other P-fractions also decrease sharply. The DOP fraction, which is generally difficult to remove, decreases from an average of 0.08 mg P/l to an average of 0.04 mg P/l. The averages are based on a total of 55 influent samples and 36 effluent samples (15 samples in period A and 21 samples in period B).

It is important to note that, when calculating P-fractions, the measurement value as shown on the display of the Hach Lange spectrophotometer has been calculated. If the measured value was < 0.05 mg P/l (detection limit), then a value between 0 and 0.05 mg P/l was still given. These values were used in the calculation of the P-fractions. The calculated P-fractions are therefore indicative values.

Figure 17 **P fractions in influent and effluent of column 1 for periods A and B. (SOP: Suspended Organic Phosphorus, PO$_{4me}$: metal-bonded PO$_4$-P, DOP: Dissolved Organic Phosphorus)**



4.1.5 ONLINE PO_4-P MONITORING

The pilot was equipped with an online PO_4-P monitor which allowed to follow the trend of the PO_4-P levels as shown in Figure 18. However, since the legal consent is based on TP concentrations, the focus is mainly on offline TP and PO_4-P measurements with Hach Lange cuvette tests. In practice, online PO_4-P measurement will be a good method to detect breakthrough in time, but the measurement inaccuracy in the range 0.05 - 0.3 mg P/l still appears to be quite large with the analyzer used in this project, see Figure 19. Water utilities usually use online PO_4-P monitors from Hach Lange (Phosphax sc) which have a low detection limit of 0.015 mg PO_4-P/l. For reliable detection of P-breakthrough, this device is probably more suitable than the Swan analyzer used in the pilot project. It should be noted that there was too little time to properly maintain the installed analyzer with an intern.

Figure 18 Comparison between online and offline PO_4-P measurement in the influent to the pilot. The gray dotted line indicates when the online measurement is equal to the offline measurement. The further away the data points are from the gray y=x line, the greater the deviation between online and offline measurement

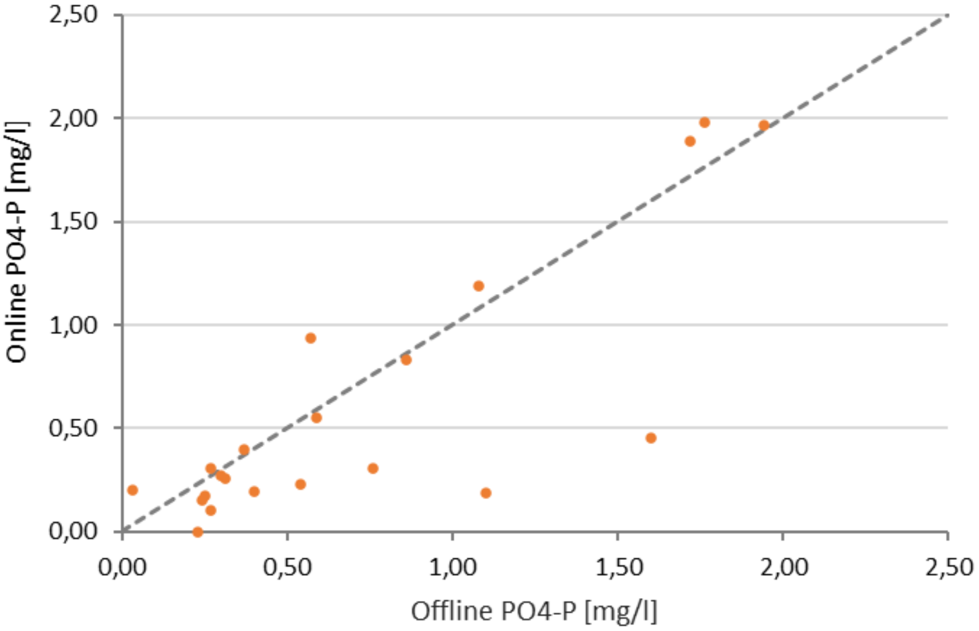
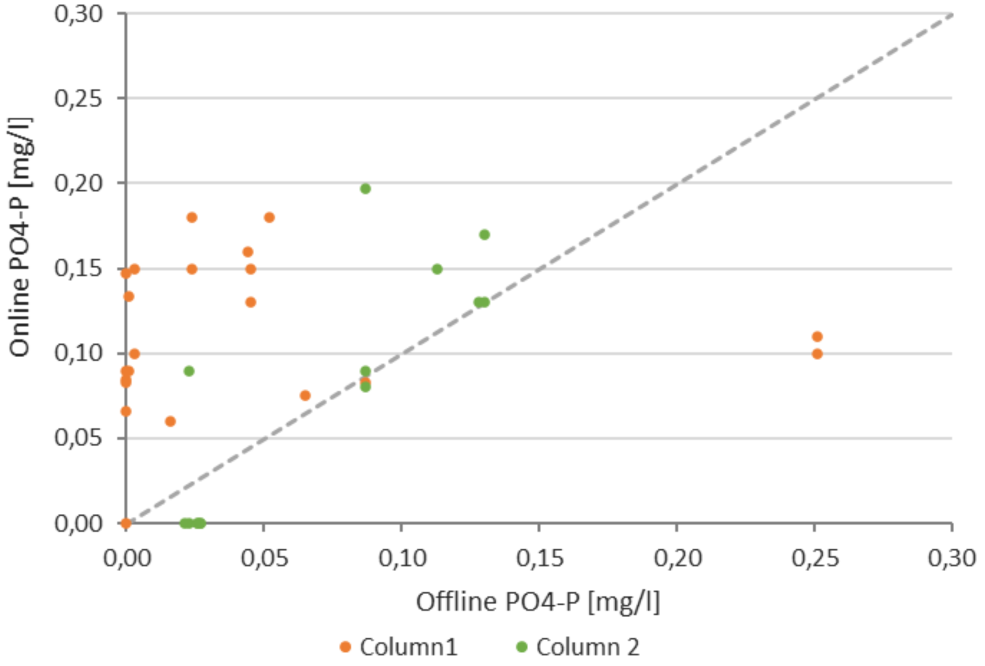


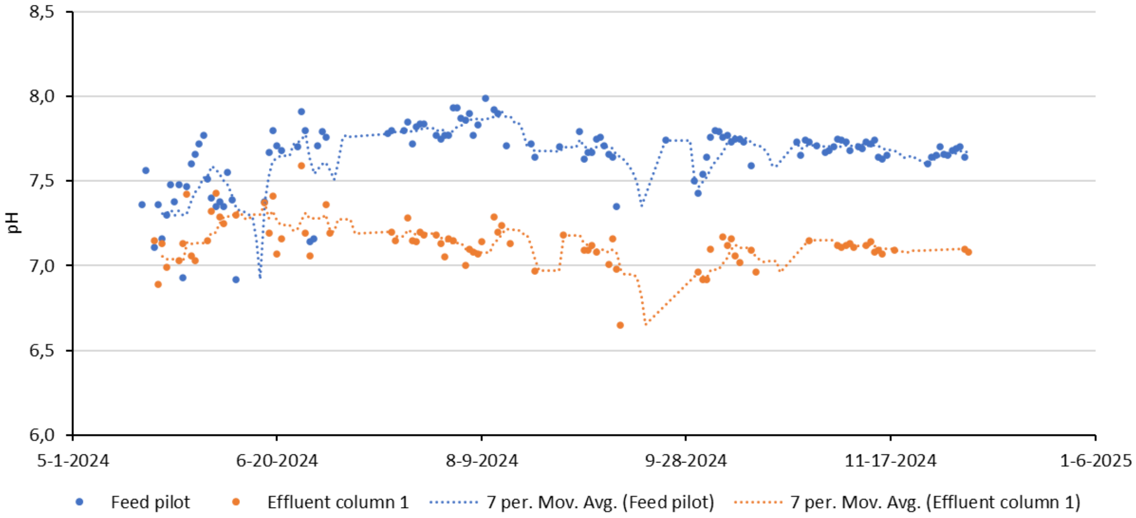
Figure 19 Comparison between online and offline PO_4-P measurement in effluent of columns 1 and 2. The gray dotted line indicates when the online measurement is equal to the offline measurement. The further away the data points are from the gray y=x line, the greater the deviation between online and offline measurement



4.1.6 PH

The pH of the influent and the effluent of the pilot were measured daily with a portable pH meter. Figure 20 shows the pH development in both streams. It is notable how the pH drops slightly comparing effluent to influent. It is not entirely clear what causes the pH drop. It is possible that part of the organic matter is degraded in the column and the CO_2 produced in this process leads to a drop in the pH. Another possible explanation is partial adsorption of bicarbonate (HCO_3^-) leading to a limited pH drop.

Figure 20 pH in influent and effluent of column 1



After regeneration, the pH after the column is temporarily high (pH 14) but quickly droping to pH 10. By recirculating the first water that comes out of the column after regeneration (with pH > 10) to the activated sludge system, a temporary pH peak in the effluent is prevented. This backwash flow rate is very limited and has no noticeable impact on the pH in the activated sludge process or P removal. To discharge the effluent, the pH does not have to be lower than pH 10 since there are several columns in parallel operation and the effluent from the column that has been regenerated is diluted with effluent from the other columns that are in normal operated.

4.1.7 REMOVAL OF OTHER COMPONENTS

The removal of COD, calcium, sulfur and heavy metals in the BiOPhree column is discussed below.

COD

In phase 1 of the pilot trial, the COD content in the effluent of the WWTP was determined for 19 days with cuvette tests in 24-hour flow proportional samples and on 17 days after columns 2 and 3 (grab samples):

- Effluent rwzi: 33.1 mg COD/l
- Effluent column 2: 26.3 mg COD/l
- Effluent column 3: 25.3 mg COD/l

On average, about 7 mg of COD/l was removed in the pilot by the sand filter and the BiOPhree column together. Part of the COD removal was due to the removal of undissolved COD (suspended solids) through the sand filter. Because suspended solids were not measured structurally, it is not clear how large this part of COD removal was. However, at the end of the pilot in phase 2, the COD removal over column 1 was determined for 12 days by taking a sample with a 5 -minute time difference before and after the column (the contact time at which the column was operated at the time). The average COD concentration in the column influent was 28.2 mg/l and in the effluent was 27.4 mg/l. In this period, column 1 removed about 0.8 mg of COD/l. The removal of dissolved COD by the iron granules therefore appears to be very limited on the basis of these measurements. The removal of undissolved COD by the sand filter was therefore approximately 6 mg/l (7 — 0.8).

COD has also been determined in various samples of the spent regenerant. This is COD that is adsorbed to the iron granules and desorbs again during regeneration ending up in the caustic soda. On average, about 1,500 mg COD/l was measured in the spent regenerant. Based on the volume of regeneration and the total amount of effluent in a cycle, this gives a COD removal in the BiOPhree column of about 3 mg/l. It is possible that the matrix of the regeneration (high pH) has influenced the measurement accuracy of the COD measurement. The calculated COD removal is therefore indicative.

HUMIC ACIDS

The spent regenerant samples of regeneration cycles 4 and 5 were determined for DOC and humic acids. The regeneration contained an average of 223 mg DOC/l, of which 80% consisted of humic acids. These humic acids can be concentrated using nanofiltration and possibly sold to agriculture and horticulture, as done by Vitens, which extracts humic acids from groundwater. A difference with humic acids from groundwater is that humic acids from effluent have a higher N content; approximately 7-8% of it is organically bound N. At a WWTP of 100,000 p.e, approximately 1 ton of humic acid/year could be recovered as a residual product of the BiOPhree process. The value of these humic acids still needs to be explored as well as whether the quality criteria for organic fertilizers can be met.

CALCIUM

The effluent from the Dronten WWTP contained an average of 53.7 mg/l of calcium. The effluent of columns 1 and 2 contained 52.9 mg Ca/l and 51.8 mg Ca/l respectively. For both columns, therefore, there was a slight calcium removal from the effluent.

Calcium was also measured in the caustic soda and rinsing water that was released after regeneration. By multiplying the amount of caustic soda and rinsing water by the measured concentrations, it was possible to calculate how much calcium was desorbed per kg of adsorbent. For column 1, this varied between 0.5 and 1.6 g Ca/kg adsorbent (average 0.94 g Ca/kg) for the last 5 regenerations.

Calcium removal by precipitation with phosphate and/or carbonate therefore takes place. Some of the removed calcium is released during regeneration, but the number of measurements has been too limited to make a good statement about any accumulation of calcium in the column.
It is important to note that, possibly, part of the calcium in the effluent from the WWTP was bound to suspended solids that were removed in the pre-filtration. As a result, the actual calcium removal in the column is lower than calculated.

SULFUR

In the samples taken in the above period, the S content[6] of the effluent from the WWTP was on average 17.1 mg/l. After column 1 and column 2, the S content was on average 16.1 and 15.7 mg/l. On average, 1 — 1.4 mg S/l was removed on these days.The mass balance established for the regenerations shows that an average of 0.8 —1.3 g S/kg is desorbed per regeneration (average 1 g S/kg).
In addition to phosphate, limited calcium and sulphate are also removed by the adsorption column.

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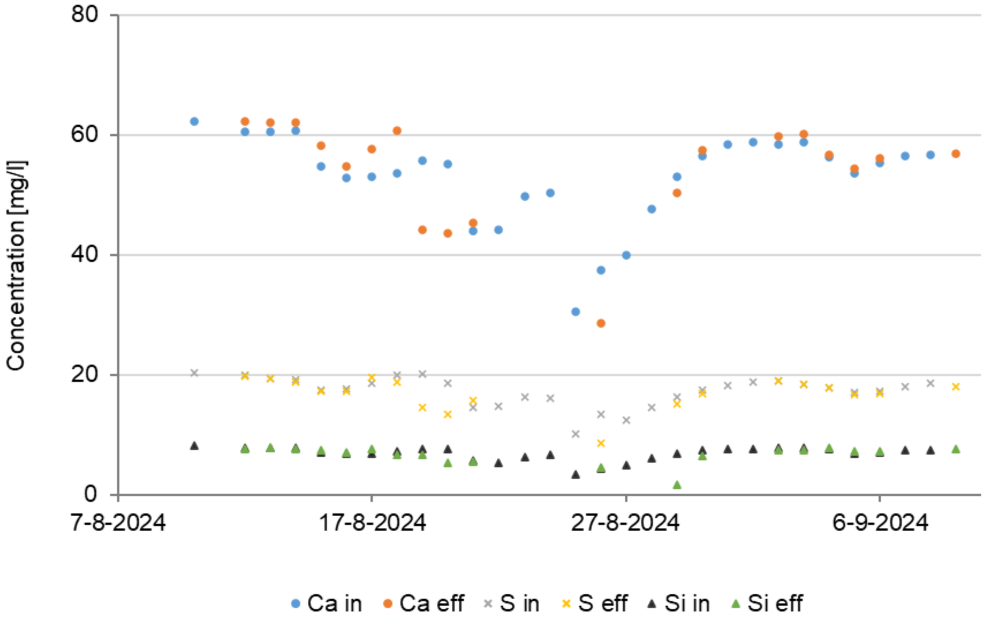
[6] With ICP-OES, only the elements are measured. Sulphur is present in effluent as SO4 but is measured as S with ICP-OES.

SILICON

The Ca, S and Si level in influent and effluent hardly differ, see Figure 21.

Figure 21 **Minerals in BiOPhree (column 1)**



The figure confirms lab results from Aquacare showing that in the first 3 cycles limited Ca, S and Si are removed and thereafter the adsorption and desorption per cycle are comparable.

HEAVY METALS

Table 6 shows the average levels of metals, sulphur and potassium in the effluent from the WWTP (pilot influent) and effluent of columns 1 and 2. The strong reduction of aluminium concentrations is striking. At the Dronten WWTP, aluminium is periodically dosed for supporting P-removal and control of the scum layer. Aluminium is known to be toxic and removal of this element from the effluent can therefore be an additional benefit of the BiOPhree technology. However, if the aluminum load in the regeneration is compared with the amount of effluent, 9-18 g of aluminum is desorbed per regeneration, which amounts to 0.02 – 0.05 mg Al/l effluent. This is a much lower reduction than shown in Table 6. Most of the aluminium in the effluent from the WWTP is probably bound to sludge particles and this fraction is removed in the pre-filtration.

It is also noticeable that zinc levels increase slightly after the column and iron levels are slightly lower than in the effluent of the WWTP. There is therefore no iron leaching in normal operation.

Table 6 **Metals, S and K in effluent WWTP (pilot influent) and effluent columns 1 and 2**

Element	Unit	Influent pilot	Effluent column 1	Effluent column 2
Al	mg/L	0.22	0.03	0.04
S	mg/L	17.08	16.10	15.67
Ca	mg/L	53.66	52.87	51.79
Zn	mg/L	0.11	0.15	0.20
Pb	mg/L	0.01		0.05
Fe	mg/L	0.22	0.04	0.06
B	mg/L	0.08	0.07	0.07
Si	mg/L	7.00	6.20	6.38
Mn	mg/L	0.13	0.11	0.14
Mg	mg/L	7.86	7.79	7.57
Cu	mg/L	0.02	0.01	0.02
Sr	mg/L	0.17	0.17	0.17
Na	mg/L	119.68	119.56	116.43
K	mg/L	32.70	28.87	28.35

4.1.8 UV TRANSMITTANCE

Because part of COD is removed in the BiOPhree column (humic acids), the clarity of the water increases slightly and with it also the UV transmittance. This can be valuable for those locations where downstream treatment is required for removal of organic micropollutants. After all, lower DOC levels lead to higher UV transmittance and/or lower O_3 consumption, which will make the removal of micropollutants more efficient.

In both phase 1 and phase 2, samples were taken on a number of days from the influent of the pilot and from the effluent of the adsorption columns. These samples were filtered with a 0.45 µm filter after which the UV transmittance was measured with a quartz cuvette at 254nm. Table 7 shows the measurement results. It can be seen that the measured UV transmittance in phase 1 is much higher than in phase 2. This is most likely caused by rainy weather and a strong dilution of the effluent. Precipitation fell on all measurement days in phase 1 except on 2 February. On this day, the transmittance is clearly lower than on the other days. Furthermore, it appears that the increase in UV transmittance in phase 1 is on average about 10% and in phase 2 only 1%. A possible explanation for this is the service life of the column. The samples in phase 1 were taken shortly after regeneration. In phase 2, the samples were taken towards the end of a cycle, just before breakthrough. In addition, the columns in phase 2 were operated with a shorter contact time. This may have led to less DOC removal and thus a more limited effect on UV transmittance.

In general, it can be said that there is a limited increase in UV transmittance.

T

able 7 **UV transmittance in pilot influent and after adsorption columns in phase 1 and 2**

Date	Phase	Influent	Effluent column 1	Effluent column 2	Effluent column3
30-1-2024	1	92%		97%	93%
02-2-2024	1	69%		96%	95%
06-2-2024	1	92%		95%	95%
07-2-2024	1	93%		94%	93%
08-2-2024	1	84%		95%	97%
09-2-2024	1	80%		93%	99%
Average		85%		95%	95%
13-12-2024	2	60%	61%		
16-12-2024	2	59%	61%		
17-12-2024	2	58%	59%		
18-12-2024	2	56%	57%		
19-12-2024	2	60%	61%		
Average		59%	60%		

4.2 RESULTS MEMBRANE TESTS FOR RECOVERY OF CAUSTIC SODA

Spent regenerant of one regeneration cycle was treated with a nanofiltration membrane (2.5'' x 40'' module) at EMI-Twente in Enschede. The purpose of this test was to determine the separation between phosphate and COD in the concentrate and the caustic soda in the permeate under different process conditions such as pressure and concentration factor.

The image below shows a photo of the regenerant (left), permeate (middle) and concentrate (right). The color difference makes it clear that the organic matter in the regeneration is largely retained and a transparent liquid is obtained that mainly contains caustic soda.

Figure 22 **Colour difference between regenerant (left), permeate (middle) and concentrate (right)**

The membrane can withstand a pressure of 40 bar and pH 14. Figure 23 and Figure 24 show the results of an experiment in which the concentrate was recirculated to the membrane's feed, causing phosphate and COD levels to increase over time. At a concentration factor of 6, the flux through the membrane drops from 42 l/m2.h to 30 l/m2.h. If the graph is extrapolated to a concentration factor of 9, then a flux of 25 l/m2.h still seems feasible.

Figure 23 Decrease in flux through the NF membrane depending on the concentration factor

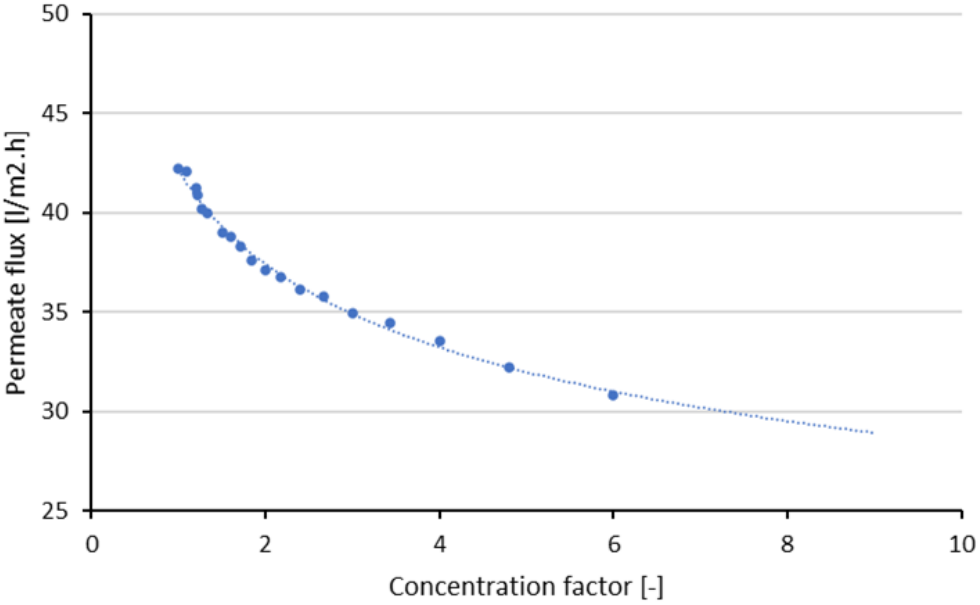
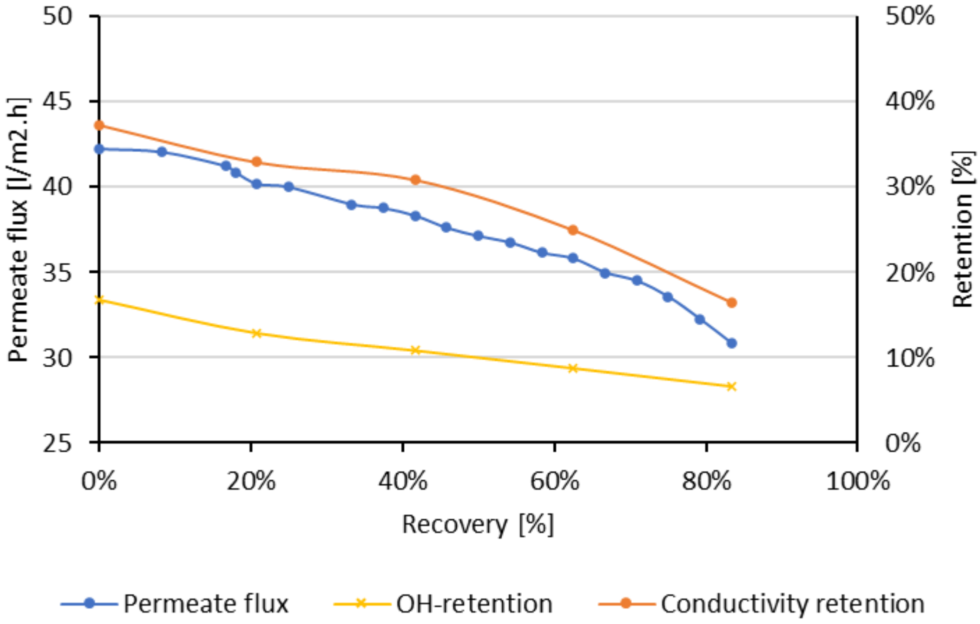


Figure 24 Flux through the NF membrane and retention of OH- and conductivity depending on recovery



The retention of OH^- decreases to < 10% with a higher recovery of the permeate (liter permeate/liter regenerate). This is beneficial because low OH^- retention combined with high permeate recovery leads to high NaOH recovery (most of OH^- ends up in the permeate). The retention of conductivity has the same trend as the permeate flux. The conductivity is determined by all ions in the water together.

In a test for 5 hours and at 38 bar, the flux was found to stabilize at 30 l/m2.h after 30 minutes, which indicates that with sufficient cross-flow (recirculation along the membrane) hardly any contamination occurred on the membrane.

The various samples taken in the membrane tests were analysed for PO_4 -P, COD and pH. From this, the removal of PO_4-P, COD and pH was calculated:

- PO_4-P: 99% removal
- COD: 88% removal
- NaOH: 2% removal

Phosphate and COD are therefore for the most part retained by the membrane and caustic soda is hardly retained. The experiments confirm that about 70 -90% of the caustic soda can be recovered from the spent regenerant.

4.3 RESULTS OF LAB RESEARCH

Parallel to the pilot tests, lab studies were also carried out. This section describes the results of column tests with effluent from WWTP Den Bosch (chapter 4.3.1), Ca precipitation tests with the spent regenerant of the pilot and the concentrate of the nanofiltration test (chapter 4.3.2) and research into combined suspended solids removal and P adsorption in the BiOPhree column (chapter 4.3.3).

4.3.1 COLUMN TESTING

Two lab columns were filled with GFH granules and fed with effluent from the Den Bosch WWTP. In each case, the column was regenerated in the same way as in the pilot. The test differed from the pilot in the following respects:

- Slightly different effluent composition, see chapter 3.5.1;
- Feeding with a constant P content per cycle;
- Higher levels of P in the influent, ranging from 2 — 5 mg P/l between cycles;
- Interrupting the cycle if P content was > 0.1 mg P/l;
- Equal amounts of caustic soda and rinsing water per regeneration.

Figure 25 shows the P adsorption of the 10 cycles performed with the 2 adsorption columns (A and B). It can be seen that the adsorption capacity remains at the same level after 10 cycles, but that column B has a higher adsorption capacity than column A. The difference may be explained by short-circuit current along the wall of the column. This is difficult to prevent on a lab scale because the wall surface is proportionally much larger than with a pilot or full-scale installation.

Figure 26 shows the mean P desorption of columns A and B as a percentage of the adsorbed

P for the 10 different cycles. On average, 95% P desorption is achieved over 10 regenerations. Up to and including the ninth cycle, an average of 99% P desorption is achieved, which indicates a very successful regeneration. In the tenth cycle, P desorption dropped to 60%. It is not clear what the cause of this is.

Figure 25 P adsorption over 10 cycles during lab column test with effluent from WWTP Den Bosch

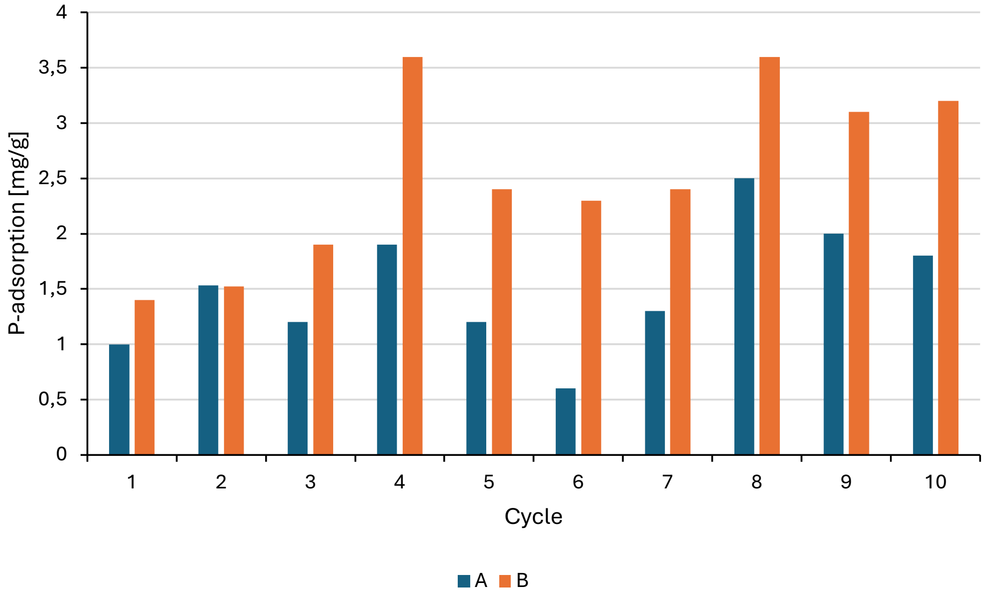
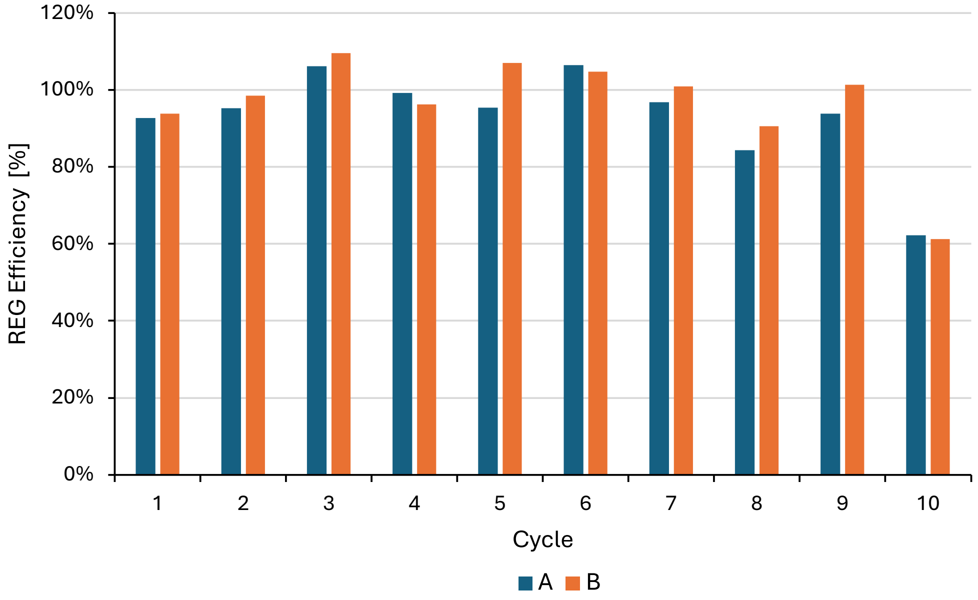


Figure 26 P desorption over 10 cycles during lab column test with effluent from WWTP Den Bosch



4.3.2 CALCIUM PRECIPITATION TESTS

Calcium phosphate precipitation tests were done with the concentrate of the NF tests and spent regenerant of one regeneration cycle of an adsorption column . The image shows that a white precipitate is formed due to calcium phosphate precipitation after adding $CaCl_2$ to the concentrate and spent regenerant. With a molar ratio of 2 mol Ca/mol P, significantly more precipitation is formed than with a molar ratio of 0.5 mol Ca/mol P.

Figure 27 **Visual representation of the results of the calcium precipitation tests**

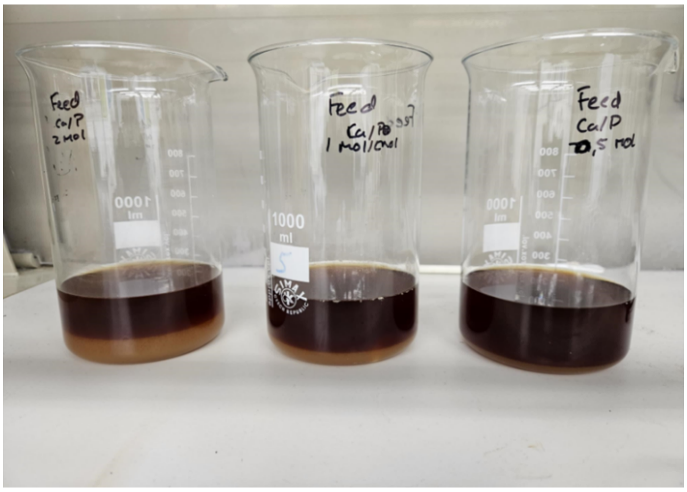


Table 8 shows a P removal of 69% - 91% at a Ca/P ratio of 2 mol/mol. The removal is somewhat higher in the spent regenerant than in the concentrate. The reason for this is not known. Theoretically, 1.7 mol Ca per mol P is needed to precipitate all phosphorus. Since the concentrate can be recirculated over the precipitation reactor after phosphate precipitation, incomplete phosphate removal is not objectionable.

Table 8 **Results Ca precipitation tests for concentrate (1418 mg P/l) and spent regenerant (354 mg P/l)**

Ca/P ratio	P Removal	
Mol	Concentrate	Regenerated
0,5	25%	33%
1	53%	56%
2	69%	91%

4.3.3 SUSPENDED SOLIDS REMOVAL IN BIOPHREE COLUMN

The pilot plant was equipped with a pre-filtration for particulate matter removal so that the adsorption column did not become clogged with suspended material from the effluent of the WWTP. The cost calculations made for the design of a practical installation (see chapter 5) showed that pre-filtration still accounts for a significant part (approximately 40%) of the investment costs of a full-scale installation.

During the project, in consultation with the supervisory committee, it was decided to explore whether the pre-filtration can be integrated with the adsorption column. This could lead to a more compact and easier installation.

In order to clarify the possibility of a combined suspended solids removal and phosphate adsorption in the BiOPhree column, the following questions must be answered:

1. Are suspended solids captured by the iron granules?
2. Can the captured suspended solids be separated from the granules by backwashing the column and what backwash speed is required for this?
3. What is the consequence of backwash for the saturation rate of the adsorbent?

To answer these questions, some exploratory lab experiments have been done, which are discussed below. The results give a first idea of the feasibility of combined suspended solids removal and P adsorption in one single column. Chapter 5 discusses possible pre-filtration techniques and the associated costs in more detail.

TSUSPENDED SOLIDS AND BACKWASH EFFECT

In a first experiment, a lab column (1 metre high[7], diameter 5 cm) was partly filled with a total of 312 grams of iron granules (GFH). The column was fed from top to bottom with effluent to which suspended solids had been added up to 50 mg/l. This was done by adding a measured amount of gravitationally thickened surplus sludge to 10 liters of effluent. Surplus sludge and effluent originated from the Amersfoort WWTP.

The effluent was pumped over the column with a flow rate of 6 l/h, which amounts to an EBCT of approximately 6 minutes, comparable to the setting at which the pilot was operated.

Result

In this experiment, the suspended solids were completely captured in the upper part of the column. Due to the SS removal, the pressure in the column slowly increased to 0.5 bar.

The column was then backwashed at a speed of 11 m/h and at a speed of 18 m/h. Five fractions of 2 liters were collected in which TSS and VSS were determined. The results of the measurements are summarized in Table 9.

Table 9 **TSS and ash content from samples taken during backwash BiOPhree for TSS Removal**

Fraction	Speed m/h	volume L	TSS mg/l	TSS mg	Ash content %
1	11	2	236	472	55%
2	11	2	28	55	60%
3	11	2	19	38	58%
4	18	2	85	171	71%
5	18	2	38	76	72%
Total		10		812	

The measurements show that a high TSS content is present in the first fraction in particular and that a lot of suspended solids are washed out. The second and third fractions contain much less TSS, which indicates that with a short backwash, a large part of the TSS can already be washed out from the column. If the backwash speed is increased to 18 m/h, the TSS content increases slightly.

The last column shows the ash content of the TSS. The data shows that the first three fractions have an ash residue of 55-60% and the last two fractions have an ash residue > 70%. The higher the ash residue, the greater the proportion of granules that leach out with sludge particles.

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[7] The bed height in this lab experiment was 30 cm. In practice, the bed height will be higher: 1 -2 m.

Based on the DS balance and the ash residue of activated sludge (approximately 20%) and iron granules (approximately 90%), it is possible to estimate how much suspended solids (sludge particles) and how many granules have been washed out. At 11 m/h, about 50% of the sludge particles are washed out and 0.1% of the granules. If the backwash speed is then increased to 18 m/h, 26% extra sludge particles and 0.06% extra iron granules are washed out.

The experiments show that sludge particles are backwashed more efficiently and there is not significant leaching of the iron granules. Howevera daily backwashing of the column can lead to a significant loss of iron granules. More tests are needed to clarify the best backwash regime when the suspended solids removal is integrated into the adsorption column.

EFFECT OF BACKWASH ON STRATIFICATION

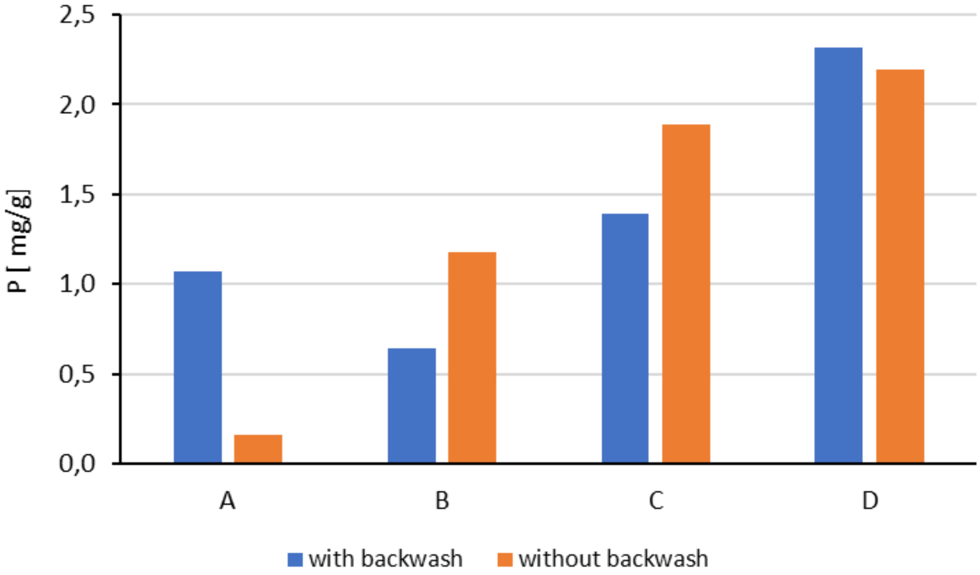
In normal operation of a BiOPhree column, the adsorbent is slowly saturated with phosphate from top to bottom, after which breakthrough occurs and regeneration is necessary. If the column is backwash daily to wash out captured suspended solids, the stratification in the column will be disrupted. The question is to what extent and how large the effect will be on the adsorption capacity. To get a first idea of this, a lab column was filled with GFH granules and then saturated with phosphate. The column was then backwashed once at a rate of 22 m/h for 5 minutes. With the help of ICP-OES analysis, the P content of the granules was determined at different heights in order to gain insight into the stratification. The measurements were made on granules saturated with P that were and were not backwashed.

Figure 28 shows the stratification in both columns. The bed is divided into four equal fractions: A to D (A: at the top of the column, D: at the bottom of the column). It is important to note that, because the column is is fed from the bottom in the lab, the granules in fraction D in principle contain the highest P content. In practice, this will be the other way around because the column will then be fed from the top.

The figure makes it clear that a single backwash leads to a partial disruption of the stratification:

- In the column that has not been backwashed, the P contents gradually increase from fraction A to D.
- This is not the case for the column that has been backwashed, and the lowest levels are measured in fraction B. Fractions C and D still show high levels, which indicates that stratification is only disturbed at the top of the grain bed.

Figure 28 P stratification in columns with and without back wash. A: upper fraction, D: lower fraction



A limited disturbance of the stratification at the top of the bed probably does not have a major influence on the breakthrough time of phosphate because this is mainly determined by the stratification at the bottom of the bed.

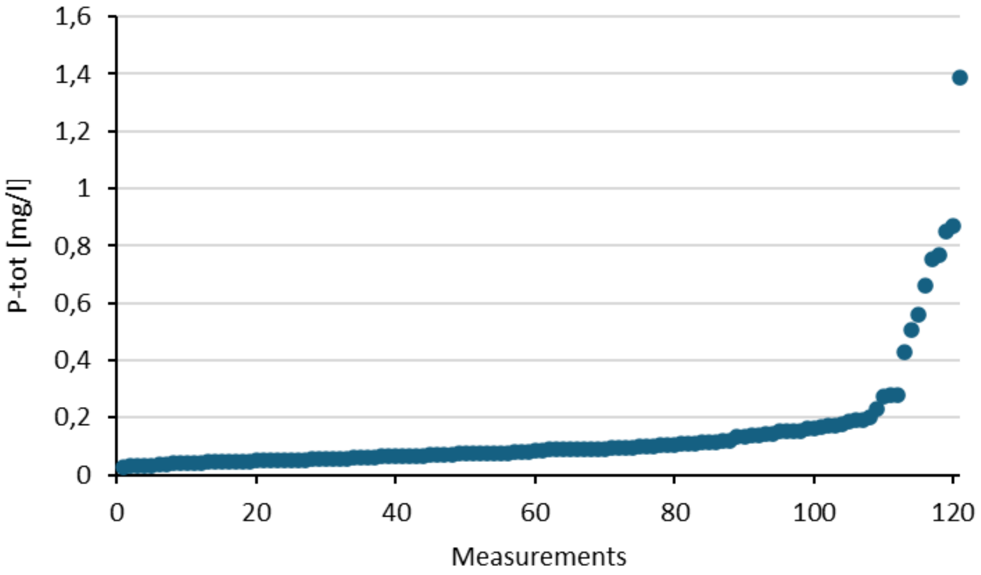
4.4 DISCUSSION

This section discusses the most important results. This includes the achievable P content after the adsorption column, the regeneration efficiency, caustic soda recovery and integration of the pre-filtration with P adsorption.

EFFLUENT P CONCENTRATION

The results described in the previous paragraphs show that the BiOPhree techno-logy is able to achieve very low P levels even after 6 regenerations and an operating time of approximately 6 months. Effluent concentrations are stable, despite variations in P levels in the influent of the pilot, which makes an average P content of approximately 0.1 mg P/l possible, even with a less constant effluent quality of the WWTP. Regeneration of the adsorbent in time is an important precondition for this because the P content in the effluent of the column increases rapidly after saturation. This is illustrated in the graph below, in which P total measurements in the effluent of column 1 are shown. The graph shows a typical hockey stick pattern caused by a small number of measurements taken after saturation of the adsorbent (read: regenerated too late). If the P contents > 0.3 mg P/l are disregarded, the mean TP concentration after the column is 0.09 mg P/l.

Figure 29 TP effluent column 1 (Hach Lange cuvette testing) from 17-6-2024 to 20-12-2024



REGENERATION

The P balances that have been drawn up for the different regeneration cycles show that the amount of caustic soda used for regeneration has a major influence on the degree of P desorption. The rinses with softened water and the neutralization of the column after regeneration are also important, although this cannot be completely proven based on the pilot data. With this optimization, an average of 82% of the adsorbed P could be desorbed in the last four regenerations.

Column tests (performed by Aquacare on a lab scale) showed an average of 95% desorption over 10 cycles for 10 adsorption/desorption cycles. In these column tests, the same regeneration recipe was applied as in the pilot, but re-generation took place always exactly after the P reached > 0.1 mg P/l in the effluent of the column. This was not the case in the pilot and more P was adsorbed until regeneration. Since regeneration was performed with the same amount of caustic soda (5 bed volumes), the pilot had to dissolve proportionally more P in the regeneration fluid. This probably led to a lower P desorption.

A better alignment between the regeneration strategy and the adsorption phase is therefore of great importance. Wetsus has demonstrated 100% P desorption in 14 cycles of adsorption/desorption on a lab scale with artificially contaminated water to which phosphate, calcium, and NOM (natural organic material) have been ad ded in similar levels as in WWTP effluent.

TYPE IRON GRANULES

Two types of iron granules were tested in the pilot; GFH and GEH. Both adsorbents gave a predominantly good P removal. During regeneration, it was found that GEH neutralizes more slowly after alkaline regeneration. The reason for this is not known. There may be a difference in porosity and this would explain why one granule is easier to neutralize than the other. The effect of neutralization after regeneration has not yet been sufficiently investigated. Based on this study, it cannot be concluded that GFH works better than GEH. For practical reasons, it has been decided to only use column 1 (GFH) for the last month, so more measurement data are available from GFH.

CAUSTIC SODA RECOVERY

The recovery of caustic soda using nanofiltration has been tested with a 2.5'' NF module in a number of short continuous tests. The results turned out to be good with a high recovery of caustic soda and high retention of COD and P. A concentration factor of 6 has been demonstrated with a stable flux. With this concentration factor and a retention of 2% NaOH, 83% of the NaOH can theoretically be recovered. The recovered caustic soda has a similar concentration as that used for re-generation; approx. 1 mol/l. In practice, it will have to be assessed which concentration factor is actually feasible. Preventing scaling (precipitation) on the membrane is essential. The higher the concentration factor, the greater the chance of scaling.

PRE-FILTRATION

Pre-filtration before the adsorption column is necessary to prevent clogging. Integration of the pre-filtration with the adsorption column has the advantage of saving one filtration step. Some exploratory tests show that it is possible to capture suspended solids in the adsorption column and that this suspended matter can also be separated again with a backwash. However, the development of a backwashing regime without loss of iron granules requires further research. Chapter 5 discusses the various possible pre-filtration techniques in more detail.

5

APPLICATION OF BIOPHREE IN PRACTICE

This chapter discusses the application of BiOPhree in practice. The process design is explained in chapter 5.1. Subsequently, chapters 5.2 and 5.4 discuss the business case and the sustainability of BiOPhree in comparison with the reference techniques. Chapter 5.5 discusses the most important points for attention for applica

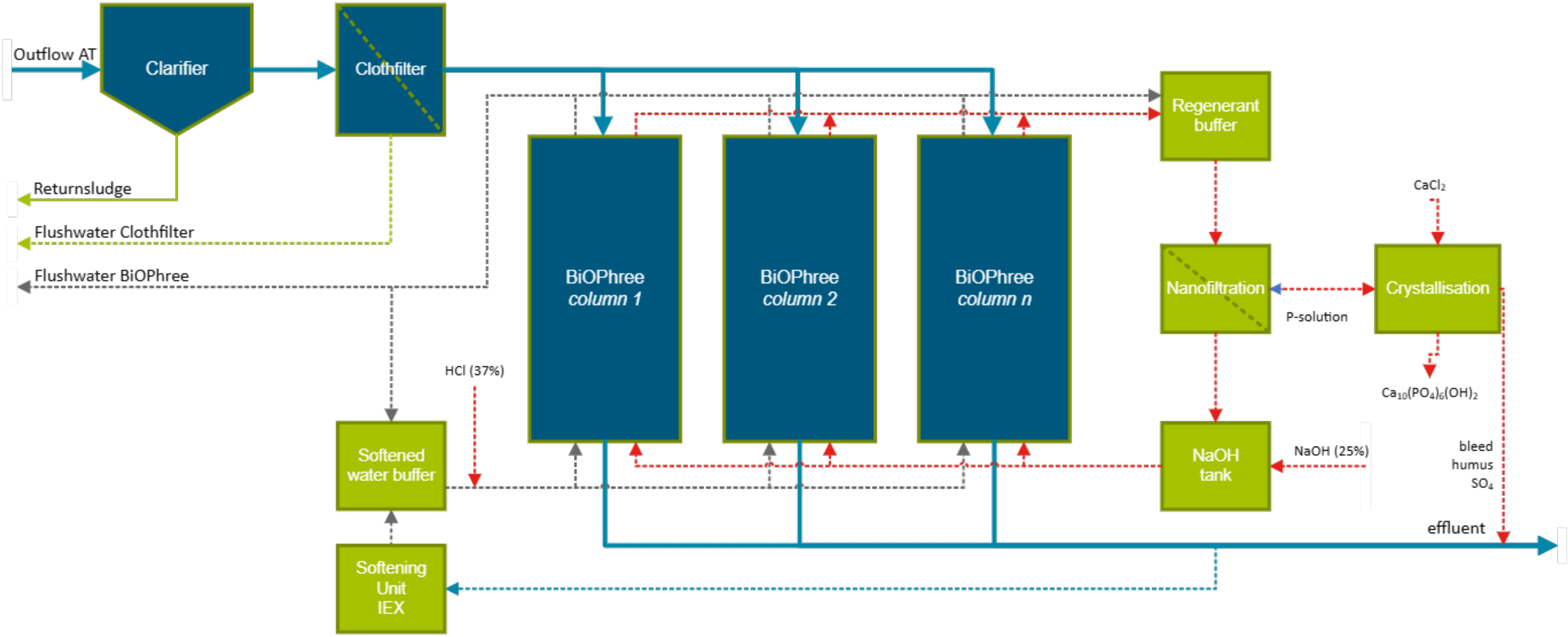
tion.

5.1 PROCESS DESIGN

Figure 30 shows a process diagram of the BiOPhree process. The process steps below are followed to remove phosphate to low concentrations:

1. **Pre-filtration** for removal of suspended solids from effluent from a WWTP. The pre-filtration can be carried out with different types of filtration techniques such as cloth filtration or a flexbed filter (formerly fuzzy filter). Dosing of a metal salt (coagulant) is not necessary because the filter only removes suspended solids.
2. **Phosphate adsorption to iron granules**. The filtered effluent is then passed over an adsorption column containing iron granules that adsorb dissolved phosphate. The contact time in the column is approximately 5 minutes. By choosing multiple parallel columns, it is possible to simultaneously regenerate 1 column and keep the others in operation. The number of columns depends on the scale of the installation.
3. **Discharge of the treated effluent**. The treated effluent (with P levels < 0.1 mg P/l) is then discharged to the surface water.

Figure 30 **Process diagram BiOPhree installation**



REGENERATION

Over time, the adsorbent becomes saturated and the P content in the effluent of the column increases. Typically, this moment is reached after about 3,000 bed volumes or after an adsorption capacity of about 4 g P/kg. By online measurement of PO_4-P in the outlet of the adsorption column, the exact moment of breakthrough can be registered. If the adsorbent is saturated with phosphate, regeneration must take place. The following steps are taken:

4. **Regeneration** with approx. 5 bed volumes caustic soda. The adsorbed phosphate desorbs and dissolves in the caustic soda. Regeneration takes place at pH 14.
5. **Rinsing of the column after regeneration** is necessary to lower the pH. The rinse water is pumped back to the beginning of the treatment process. Due to the limited quantity, this does not affect the activated sludge process.

The column is then regenerated and can be reloaded with effluent in the next cycle.

RECOVERY OF CHEMICALS AND PHOSPHATE

An important part of the BiOPhree concept is the recovery of the used caustic soda and the phosphate removed. The following steps are taken:

6. **Nanofiltration of the regenerate used.** By passing the spent regenerant (caustic soda) in which phosphate is dissolved over a nanofiltration membrane, NaOH is separated from the dissolved phosphate. NaOH ends up in the permeate and phosphate in the concentrate. The permeate can be reused immediately in a subsequent regeneration. The concentrate contains the removed P and is suitable for P recovery. This can be done centrally in a rejection water treatment (usually struvite precipitation) or decentrally at the WWTP itself by adding calcium and precipitating calcium phosphate and removing it.
7. **Phosphate precipitation.** The concentrate (about 10% of the regenerant volume) contains mainly phosphate, humic acids and sulphate. By dosing calcium to the concentrate, the phosphate can be precipitated as hydroxyapatite, which in principle can be sold to a fertilizer supplier. Another option is to transport the concentrate to an existing struvite installation and recover it as struvite. For a WWTP of 100,000 IU, this amounts to approximately 1,300 m3/y (36 loads/y).

DIMENSIONING BIOPHREE FOR A WWTP OF 100,000 P.E.

The above process design has been calculated for a WWTP with a capacity of 100,000 p.e. and two different scenarios:

- Scenario A: P requirement of 0.3 mg P/l (annual average)
- Scenario B: P requirement of 0.1 mg P/l (annual average)

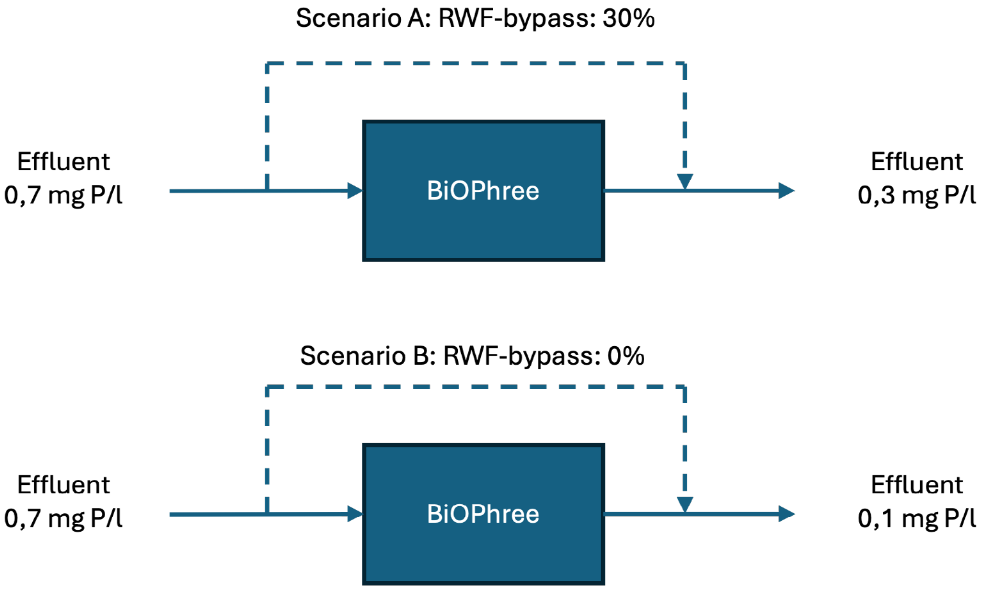
The following assumptions have been made for the effluent flow rate and composition:

Table 10 **Effluent data for process design WWTP 100,000 p.e.**

Parameter	Unit	Value
Capacity	$p.e._{150}$	100,000
Flow	m^3/d	20,000
Dry weather flow	m^3/h	625
Wet weather flow	m^3/h	3,750
Effluent composition		
P total	mg/l	0.70
DOP	mg/l	0.10
P-ss	mg/l	0.25
PO_4-P	mg/l	0.35
Suspended solids	mg/l	7

Because the effluent from the BiOPhree installation has a P content of 0.1 mg P/l, treatment of 70% of the effluent is sufficient in scenario A. In scenario B, 100% of the effluent (including the wet weather flow, WWF) will have to be treated, see figure below. The hydraulic capacity of the BiOPhree plant differs greatly for both scenarios: 1.2 x dry weather flow (DWF) for scenario A and 5 x DWF for scenario B.

Figure 31 **Post-treatment with BiOPhree for 0.3 mg P/L and 0.1 mg P/L**



It is important to note that, in practice, it will be necessary to calculate for each location which hydraulic capacity of the post-treatment is required to meet the set P requirement. Each WWTP has a different effluent quality and a different DWF/WWF ratio, which has consequences for the design of a BiOPhree installation.

Table 11 summarizes the dimensioning of the BiOPhree installation for both scenarios:

Table 11 **Dimentions BiOPhree 100,000 p.e. for 0.3 mg P/l (A) and 0.1 mg P/l (B)**

Parameter	Unit	Value	
Scenario		A	B
P requitement	mg/L	0.3	0.1
Flow to BiOPhree	m^3/h	750	3,750
Pre-filtration[1]	m^2	95	470
Bed volume	m^3	63	313
Columns	#	6	27
NaOH tank	m^3	10	10
Acid tank	m^3	1	1
NaOH dilution Tank	m^3	60	60
Effluent tank (for backwash)	m^3	50	50
Nanofiltration	m^2	58	58
Spent regenerant tank	m^3	80	80
Calcium phosphate sto-rage	m^3	1	1

[1] filtration surface

SPACE REQUIREMENTS

The space requirements for a BiOPhree installation is of course determined by the flow rate to be treated (the greater the capacity of the installation, the more space required) but also by the components required for regeneration; storage of caustic soda, rinse water tanks, etc. The required surface area has been estimated for both scenarios and varies from approximately 500 m^2 for scenario A to approximately 1,000 m^2 for scenario B. Margins for the supply of chemicals and the removal ofphosphate have been taken into account.

5.2 BUSINESS CASE

For both scenarios, the investment (CAPEX) and operational costs (OPEX) have been calculated. The following principles have been followed:

Table 12 **Financial assumptions business case calculation BiOPhree 100,000 p.e.**

Parameter	Unit	Value
Electricity	EUR/kWh	0.20
NaOH 32%	EUR/ton product	230
$FeCl_3$ 40%	EUR/ton product	235
Sludge disposal	EUR/ton	90
Management	EUR/fte	60,000
Fe granule	Euro/kg	8.00
CaP	Euro/kg P	0.50
$CaCl_2$	Euro/kg	0.75
HCl 30%	Euro/ton product	88
Maintenance	% of construction costs	2%
Depreciation	y	20
Interest	%	4%
Markup factor		1.8

The investment costs have been estimated according to the cost estimate classification system methodology.

The costs for scenario A and B are summarized below:

Table 13 **Cost of scenario A (0.3 mg P/l) and scenario B (0.1 mg P/l)**

Parameter	Unit	Scenario A		Scenario B	
Electricity	Euro/year	€	36,000	€	53,000
Sludge production	Euro/year	€	12,000	€	17,000
Caustic soda[1]	Euro/year	€	41,000	€	61,000
Maintenance	Euro/year	€	146,000	€	448,000
Labour	Euro/year	€	30,000	€	30,000
Others (not specified)	Euro/year	€	63,000	€	329,000
Total OPEX	Euro/year	€	328,000	€	938,000
OPEX per m^3	Euro/m3	€	0.04	€	0.13
Investment	Euro	€	13,100,000	€	40,300,000
CAPEX per m^3	Euro/m^3	€	0.13	€	0.41
Total[2] per m^3	Euro/m^3	€	0.18	€	0.53
Total per kg P removed	Euro/kg P	€	443	€	892

[1] 70% NaOH recovery has been assumed

[2] This is the cost price calculated per m^3 of wastewater that is processed at the WWTP.

The following conclusions can be made:

- The cost for scenario A is 0.18 EUR/m^3. This is comparable to reference techniques such as sand filtration and cloth filtration (0.16 -0.22 euros/m^3) that remove phosphate from effluent using metal salt dosing.
- The cost for scenario B (0.1 mg P/l) is considerably higher than for scenario A, which is mainly due to a much higher investment; 40 M euros instead of 13 M euros. The high investment is determined by the high flow rate for which the BiOPhree must be designed to meet a P requirement of 0.1 mg P/l at all times. At 3,750 m^3/h, this flow rate is 5 times higher than in scenario A.
- At 443 euros/kg P removed, the cost per kg P removed is slightly higher than the cost for cloth and sand filtration with $FeCl_3$ dosing in STOWA 2024 -27 (250-300 EUR/kg P removed). The reason is that in the STOWA study, a higher P content was chosen in the influent of the post-treatment (0.9 mg P/l instead of 0.7 mg P/l), which means that more P is removed per m^3 of effluent. The cost is then lower because it is mainly determined by capital costs, which are independent of the incoming P content.

For scenario B (0.1 mg P/l), there are currently no alternatives to which BiOPhree can be compared. In theory, nanofiltration could be used to remove P from all the effluent to 0.1 mg P/l, whereby the concentrate still has to be treated with a sand or cloth filter for P removal using metal salts. To the best of our knowledge, there is no practical experience with this combination of techniques, which makes it challenging to make a reliable cost estimate.

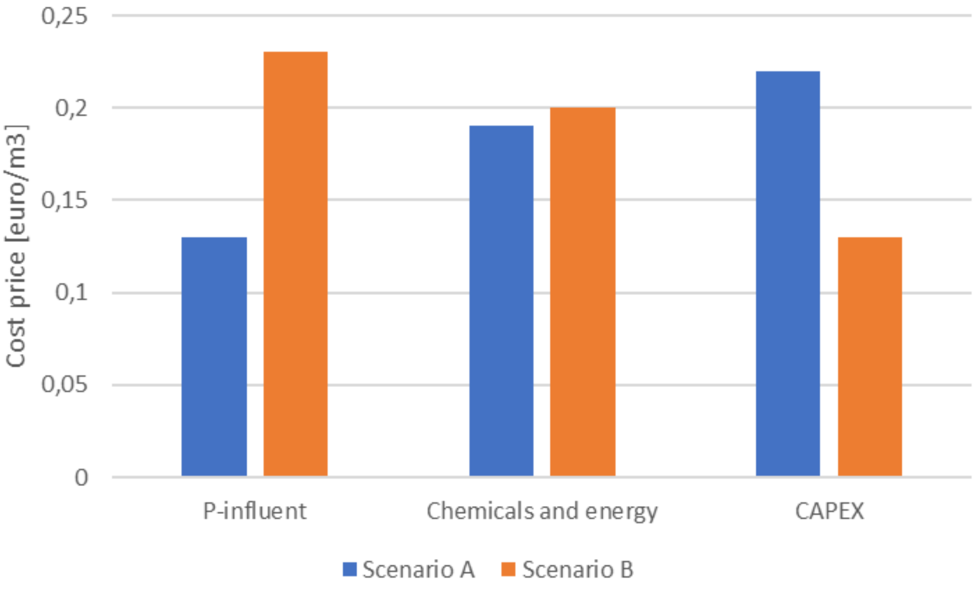
5.3 SENSITIVITY ANALYSIS

Fi-gure 32 shows the cost of BiOPhree for scenario A (P requirement 0.3 mg P/l) de pending on the P concentration in the influent of the plant (0.5 mg/l or 0.9 mg/l), the chemicals and energy price (respectively 2 and 3 times higher than in scenario A) and the CAPEX of the plant (+30% or -30% compared to scenario A).

The cost of BiOPhree appears to be highly dependent on the incoming P concentration and the investment costs of the installation and much less on the price of chemicals and energy.

Optimization of phosphate removal in the WWTP is therefore very important to keep the costs of post-treatment low. Through a good design in which the adsorption columns and the regeneration are optimally coordinated, the investment costs can be reduced significantly. It remains important to keep chemicals and energy consumption low for the lowest possible CO_2 footprint.

Figure 32 **Sensitivity of the cost of BiOPhree for P influent concentration (A: 0.5 mg P/l, B: 1 mg P/l), price of chemicals and energy (A: 2x higher, B: 3x higher than business case) and CAPEX installation (A+30%, B: -30%). Effluent P-requirement: 0.3 mg P/l.**



5.4 SUSTAINABILITY

The sustainability of a technology can be calculated on the basis of CO2 emissions, amongst other indicators. This was done for the two scenarios for which the business case was also calculated: a WWTP of 100,000 p.e. with a P requirement of 0.3 mg P/l and a P requirement of 0.1 mg P/l.

P REQUIREMENT 0.3 MG P/L

The CO2 emissions of a 100,000 p.e. BiOPhree installation with a P requirement of 0.3 mg P/l amount to approximately 136 ton of CO2/y (19 g CO2/m3)[8]. 7 This is comparable to the CO_2 emissions of reference techniques such as cloth and sand filtration (18-22 g CO_2/m^3). 80% of CO_2 emissions are determined by energy and caustic soda consumption, whereby a NaOH recovery of 70% is assumed. CO_2 emissions can be further reduced by higher NaOH recovery and optimal pre-filtration.

P REQUIREMENT 0.1 MG P/L

If a 100,000 p.e. WWTP has to meet a P requirement of 0.1 mg P/l, then all the effluent must be treated and the adsorbent has to be regenerated more often. CO_2 emissions then rise to 36 g CO_2/m^3. It is not possible to calculate a reference scenario for this option.

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[8] If the CO2 emissions per m^3 are compared with the average CO_2 emissions related to wastewater treatment (278 g CO_2/m3, Waves database 2023), then the increase due to the application of BiOPhree and other post-treatment techniques for far-reaching P removal is therefore about 7%.

OTHER SUSTAINABILITY ASPECTS

Phosphate recovery

Because the extra phosphate removed is recovered as calcium phosphate in a BiOPhree system, this technology is considered more circular than reference techniques that depend on metal salt dosing.

Humic acids recoveryHumusterugwinning

Because a BiOPhree also removes part of the humic acids from the effluent, their recovery could be of interest. For a WWTP of 100,000 p.e., it has been calculated that about 1 ton of humic acid can be recovered per year. Research has yet to show whether the composition is suitable for use as organic matter fertilizer in agriculture and horticulture.

Sludge production

Because no metal salt is dosed with BiOPhree for P-removal, the extra sludge production is lower than with a sand/cloth filter. In scenario A, this saves more than 70 ton DS/year in sludge production (320 ton of sludge cake/year). The percentage increase in sludge production due to effluent post-treatment is about 2% for BiOPhree and about 7% for cloth filtration.

5.5 DISCUSSION

The results of the lab and pilot studies show that BiOPhree is suitable to achieve P levels of 0.1 mg P/l, which is significantly better than reference techniques such as sand and cloth filters that remove phosphate to 0.25 – 0.28 mg P/l (STOWA 2024-27, UKWIR (2018), The National Chemical Investigations Programme 2015-2020).

However, in order to discharge effluent with 0.1 mg P/l at all times, it is necessary to treat the entire flow rate in WWF. The business case calculations show that the costs will then be > 0.5 EUR/m^3. Therefora, in practice, a post-treatment that can treat approximately twice the DWF is preferred, so that on an annual basis approximately 80% of the effluent is treated and an average effluent quality of 0.4 mg P/l is achieved (see: STOWA 2024-27).

The costs and CO2 emissions of BiOPhree in this scenario are comparable to a downstream cloth filter. The added value of BiOPhree compared to cloth filtration, considering both techniques have similar costs and comparable CO_2 emissions, is discussed below.

P REMOVAL

During DWF, BiOPhree can achieve 0.1 mg P/l, whereas a cloth filter achieves 0.25-0.28 mg P/l. Although in the comparison in the business case an annual average P concentration of 0.3 mg P/l is achieved with cloth filter treating 90% of the yearly flow and BiOPhree 70%, better quality during DWF is beneficial for the water system. Because DWF occurs approximately 75%[9] of the time, a WWTP with a BiOPhree post-treatment can discharge effluent that already meets the WFD standards to the water system most of the time (often this is 0.11 mg P/l for phosphate). This does not apply to a cloth or sand filter. Especially in water bodies where the phosphate content is strongly influenced by WWTP effluent, BiOPhree can therefore be of added value due to the low P levels.

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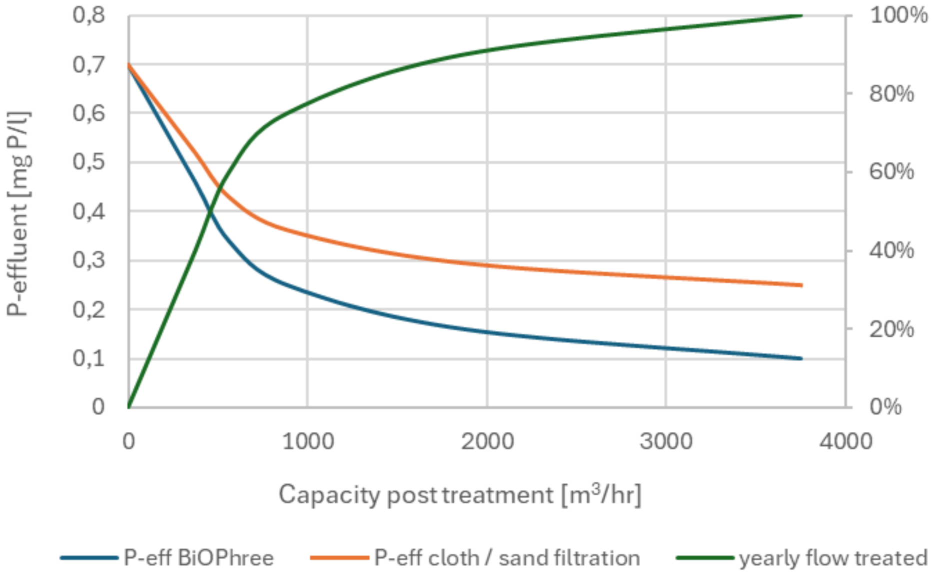
[9] It rains about 10% of the time in the Netherlands, but about 25% of the time there is an increased supply flow due to precipitation.

WATER FRAMEWORK DIRECTIVE STANDARDS

For the Water Framework Directive (WFD), a summer requirement for nutrients applies based on an average of 6 measurements in the summer half of the year at the WFD measuring point in the water system. The requirement for phosphate varies from 0.04 – 0.75 mg P/l (average 0.11 mg P/l). The requirement depends on the type of surface water and its susceptibility to eutrophication. Stagnant water is generally more susceptible to eutrophication than running water.

If a BiOPhree installation is expanded so that more water can also be treated during WWF, it is possible to get the annual average P content well below 0.3 mg P/l. For sand and cloth filtration, 0.25 mg P/l is close to the limit of the technology. Figure 33 shows how the effluent P content depends on the capacity of the post-treatment and the treated annual flow rate for BiOPhree and cloth/sand filtration. As more effluent is treated in that step, the P content naturally decreases and the difference between BiOPhree and cloth/sand filtration increases.

Figure 33 **Comparison of P effluent BiOPhree and cloth filtration depending on the capacity (m^3/h) for 100,000 p.e. (DWF: 625 m^3/h, P effluent WWTP: 0.7 mg P/l)**



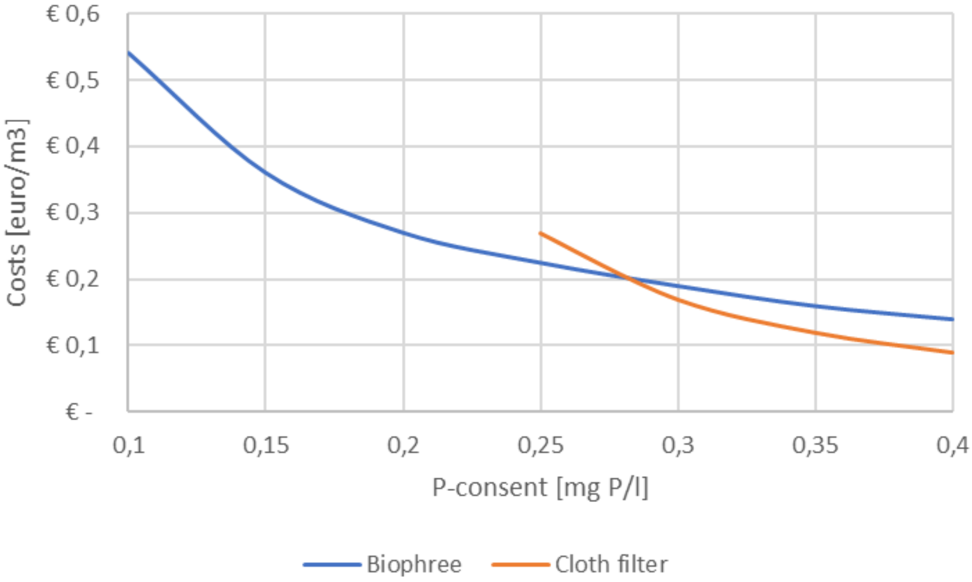
The DWF in the example above is 625 m^3/h. If the post-treatment is designed at three times the DWF (approximately 2,000 m^3/h), BiOPhree already achieves a P concentration of 0.15 mg P/l compared to 0.3 mg P/l for cloth/sand filtration. With BiOPhree, it is therefore possible to make progress in terms of P removal, provided that more effluent is post-treated.

A possible additional advantage of BiOPhree is the very good smoothing of P peaks in the influent. The pilot showed that with strongly fluctuating P concentrations in the influent, the P level in the effluent can always be < 0.1 mg P/l if regeneration takes place in time. In order to properly smooth out peaks in the influent of cloth/sand filtration, a high metal salt dosage is required, combined with tight monitoring to provide timely adjustments in the dose. The disadvantage of a high dosage is that the flocculation due to iron phosphate and iron hydroxide increases sharply and the filter has to be backwashed more often. This is not the case with BiOPhree, which makes this technology better able to smooth out peaks.

COST

As the business case shows, the costs of post-treatment increase with a stricter P requirement. Figure 34 presents this efefct for a P requirement ranging between 0.1 — 0.4 mg P/l.

Figure 34 **Cost of BiOPhree and cloth filtration depends on the effluent P requirement for a WWTP of 100,000 p.e.**



The figure shows that with a more flexible P requirement (> 0.28 mg P/l), cloth filtration is cheaper than BiOPhree. With a stricter P requirement, BiOPhree be comes cheaper and with a P requirement < 0.25 mg P/l, BiOPhree is the only op-tion. The costs of BiOPhree have been calculated with the knowledge that is now available about the technology based on the pilot and lab research. Experience with technology development shows that costs can still reduce sharply through optimizations, which makes this estimate conservative. A number of possible optimisations are briefly explained below with an estimate of the impact on the cost.

P concentrations after secondary sludge settling

The P concentrations after the post-sedimentation tank (i.e. in the supply to the post-treatment) is a very important parameter that has large influence on the costs. With a higher P content after the post-sedimentation tank, more effluent will have to be treated in order to meet a certain discharge standard. The installation must therefore be designed for a larger flow and the investment costs will increase. This applies to both BiOPhree and cloth/sand filtration. For BiOPhree, there is also the fact that caustic soda consumption increases with higher P levels in the supply because more regeneration is required. Conversely, an optimized activated sludge process with good P removal will lead to lower post-treatment costs. This has also been highlighted in the STOWA report on WFD measures (STOWA 2024-27). A P content of 0.5 or 0.9 mg P/l in the supply results in treatment costs of 0.14 EUR/m^3 and 0.24 EUR/m^3 respectively. An optimized activated sludge process is therefore essential for cost-effective post-treatment.

Integrated suspended solids removal

In the business case in chapter 5.2, a cloth filter has been assumed for the pre-filtration before a BiOPhree installation. The costs of the cloth filter are derived from cost estimates of other projects at Dutch water utilities where a cloth filter will be installed. However, experience with cloth filtration in the Netherlands is limited (in 2025, only one practical installation will be operational). This project did not look at the effect of different pre-filtration techniques on the costs. It is possible that other techniques, such as the Flexbed filter (Bosman Water), reduce the costs. This may also apply to the integration of suspended solids removal in the adsorption column, see 4.3.3. Since the costs are now determined for approximately 30% by the CAPEX of the pre-filtration, it is expected that by optimizing the pre-filtration, the treatment costs of BioPhree can be significantly reduced from 0.19 EUR/m^3 to approximately 0.14 EUR/m^3.

Lower contact time

In the last month of phase 2, the pilot was operated on an EBCT of 5 minutes. Suppliers of Fe granules indicate that an EBCT of 3 minutes should certainly be possible. In the pilot study, there was no time to also test this shorter EBCT. A shorter contact time will lead to a smaller column, which can further reduce the investment costs to approximately 0.13 EUR/m^3.

Development of iron granules

In the pilot study, various iron granules were tested as offered by suppliers for the removal of arsenic from groundwater. The use of these iron granules for phosphate removal is a new application that can probably be improved if the pro-duction of the iron granules is specifically focused on suitable properties for P adsorption. In this case. an optimal grain size with minimal bed resistance and a high specific surface area are important. Although the results of this pilot show that there are materials on the market with which the technique works excellently, Aquacare and Haskoning are discussing with suppliers possibilities to make the iron granules even more suitable for reaching low P concentrations in WWTP effluent

Other factors

NaOH-recovery

Caustic soda consumption accounts for about 9% of the costs. Recovery through the nanofiltration membrane and loss in the column through exchange and flushing have already been calculated. Optimisations may be able to reduce caustic soda consumption even further so that caustic soda costs are lower.

P-recovery

The P recovery from the concentrate was based on a crystallisation process at the WWTP where the BiOPhree installation is located. If there is a struvite installation within a short distance, it is probably more cost-effective to transport the concentrate and treat it together with the reject water for struvite production. The best method of phosphate recovery will have to be assessed for each location.

In summary, it can be said that there are still many opportunities to reduce the cost price of BiOPhree, making the technology even more cost-effective than the business case already shows. It is important to note that theoptimizations described above are not yet included in the cost price of BiOPhree in Table 13.

The comparison between BiOPhree and sand/cloth filtration is summarized in table 14 with scoring of the most important criteria.

Chloridege content effluent WWTP

A disadvantage of conventional post-treatment techniques for extensive P removal is that the dosage of $FeCl_3$ increases the chloride content in the effluent. Depending on the metal to P ratio, this can be as much as 10 -20 mg/l of additional chloride in the effluent. High chloride levels are generally undesirable in sensitive surface water (van der Molen et al, 2006). An advantage of BioPhree is that no metal salts are dosed and the chloride content in the effluent is not affected.

Table 14 **Summary comparison of BiOPhree with sand/cloth filtration for different criteria**

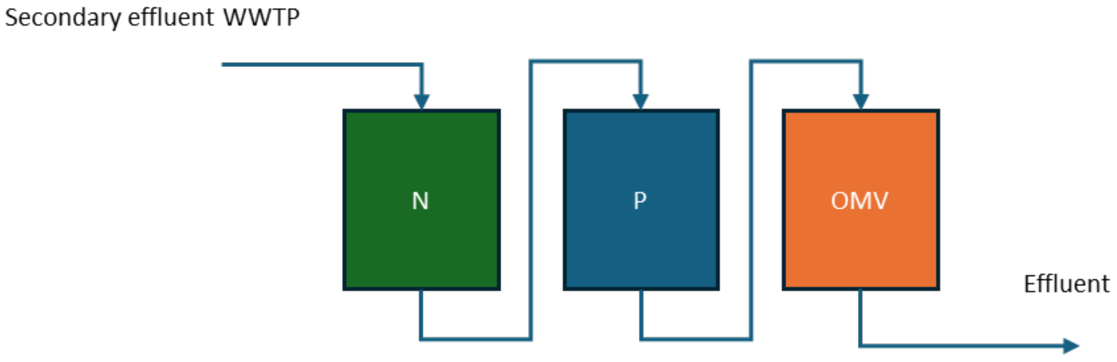
Criteria	BiOPhree	Explanation
P Removal	+++	BiOPhree can achieve lower levels of P, for example by good smoothing of influent peaks and partial DOP removal. This is advantageous for DWF, but with a larger capacity of the post-treatment also for WWF.
Costs	+	BiOPhree is already comparable to cloth/sand filtration in terms of costs, but can become even more cost-effective through optimization.
Durability	++	The CO_2 emissions of BiOPhree are comparable to those of sand/cloth filtration. With BiOPhree, much less sludge is produced and the removed phosphate can be recovered. It may also be possible to recover humic acids.
Flexibility	++	BiOPhree can be expanded for a higher flow rate to remove more P. With sand/cloth filtration his is only possible to a limited extent because a relatively large amount of effluent must be treated to meet a P-requirement of 0.3 mg P/l.

COMBINATION OF BIOPHREE WITH OTHER POST-TREATMENT TECHNIQUES

In addition to strict requirements for phosphate, some WWTPs also have strict requirements for nitrogen and organic micropollutants. The most logical order for achieving all these effluent requirements most efficiently is:

1. Removal of nitrogen.
2. Removal of phosphate.
3. Removal of organic micropollutants.

Figure 35 **Post-treatment train for removal of N, P and organic micropollutants**



The following points are highlighted:

- Depending on the receiving surface water, the requirement for N, P and organic micropollutants will be more or less strict.
- For nutrients, the “one in all in” principle applies in the WFD, i.e. if the ecological goals can be achieved with only low P levels, then the requirements for N no longer have to be met.
- Organic micropollutants are not yet subject to a WFD requirement, but EU legislation is expected. This in itself is a reason to take this step last, because the EU requirements will come into force later than the WFD requirements.
- Low N levels require a denitrifying filter that should not become limited in P. N removal must therefore precede P removal if there is a strict requirement for both parameters.
- The P removal with BiOPhree probably no longer needs pre-filtration because the denitri-

fying filter also captures suspended solids. This is not entirely certain because a denitrifying filter can also release biomass, so a limited pre-filtration may still be needed.
- As a final step, organic micropollutants can be removed by a combination of adsorption and oxidation. Because the effluent is free of suspended matter and humic acids have been partially removed, this step will be cheaper compared to a situation in which no upstream N/P removal takes place.
- Combined removal of N, P and organic micropollutants in 1 filter is possible with the 1 -step filter, see STOWA 2009-34. However, if very low P levels have to be achieved (< 0.25 mg P/l), this technique is not an option.

6

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The following conclusions can be drawn from the pilot and lab studies of the BiOPhree technology for P removal from effluent to very low levels:

- P-removal up to 0.1 mg P/l is possible, provided that regeneration takes place in time. After 6-10 regenerations, P removal remains stable at a high level with an average adsorption capacity of about 4 g P/kg and about 3,000 bed volumes up to breakthrough (P >0.1 mg P/l). This corresponds to a runtime of about 1-2 weeks until phosphate breakthrough. At low influent P levels (< 0.5 mg P/l), the runtime can reach up to 6,000 bed volumes.
- The calculated P desorption over 4 regeneration cycles was on average 82% in the pilot. On a lab scale an average P desorption of 95% could be achieved with effluent from WWTP Den Bosch in 10 regenerations with similar conditions.
- Analysis of PO4 and dissolved and suspended P total showed that the DOP fraction decreased by about 50% over the BiOPhree column.
- In addition to phosphate, 7 mg/l COD is removed, of which 1 -3 mg/l is dissolved COD. This slightly increases the UV transmittance of the effluent light, which is beneficial for a possible downstream removal of organic micropollutants.
- The removed dissolved COD consists mainly of humic acids which may be recovered separately. For a WWTP of 100,000 p.e., this would amount to about 1 ton of humic acid/year.
- 70-90% of the caustic soda used in regeneration can be recovered using nanofiltration.
- The removed phosphate can be recovered as hydroxyapatite after calcium dosing to the concentrate. Lab tests showed 70-90% P precipitation at a ratio of 2 mol Ca/mol P. An alternative to hydroxyapatite is recovery as struvite by transporting the concentrate to a nearby struvite plant.
- For a WWTP of 100,000 IU where the effluent P content must drop from 0.7 mg P/l to 0.3 mg P/l, the treatment costs are estimated at 0.18 euros/m3 effluent. The CO2 emission of the BiOPhree technology is approximately 17 g CO2/m3 ef-fluent and depends on the P-load to be removed. If more P has to be removed, the CO2 emissions per m3 will increase and vice versa.
- Integration of the pre-filtration (for suspended solids removal) into the BiOPhree column seems possible on the basis of some exploratory lab tests. This can significantly reduce the investment costs, but more research is still needed to determine the effect of a daily backwash of the adsorption column on the P adsorption capacity.

RECOMMENDATIONS

The following recommendations are made:

Demonstration BiOPhree

The pilot and lab results together provide sufficient basis for a step towards a demonstration of the BiOPhree technology on a practical scale in which the entire concept (treatment effluent, regeneration and caustic soda recovery) is tested on a practical scale at a suitable WWTP. It is recommended to do a demonstration project at a WWTP where P removal to very low levels has added value.

Suspended solids removal (pre-filtration)

Until now, the business case has been based on pre-filtration with a cloth filter. The costs for the cloth filter are derived from estimates from other projects, but it is unclear how accurate these estimates are because only one full-scale cloth filter has been built in the Netherlands.

In addition to cloth filtration, there are several other possible techniques with which suspended solids can be efficiently removed from effluent. One of them is the flexbed filter (formerly fuzzy filter). Follow-up research is needed to find the most cost-effective solution for suspended solids removal. This can be pre-filtration with different techniques or integrated suspended solids removal and P adsorption. Both need to be investigated further.

Surface water treatment

Water utilities sometimes treat surface water for the removal of phosphate. In principle, BiOPhree is also suitable for this purpose, but the feasibility will have to be investigated separately for each location. The location must be suitable for storing chemicals (caustic soda) and for the removal of the concentrate rich in P that is produced. It is recommended, together with water utilities, to further explore opportunities for P removal from sur-face water to very low levels with the help of BiOPhree.

7

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APPENDIX A

COST KEY FIGURES AND SUSTAINABILITY CALCULATIONS

COSTS KEY FIGURES

Parameter	Unit	Value	Source
Electricity	EUR/kWh	€ 0.20	
NaOH 32%	EUR/ton product	€ 230.00	Vivochem/Brenntag, quote Jan 2023
$FeCl_3$ 40%	EUR/ton product	€ 235.00	Vivochem, quote Jan 2023
Sludge dispo-sal	EUR/ton	€ 90.00	
Management	EUR/fte	€ 60,000.00	
Fe granule	EUR /kg	€ 8.00	Supplier information
CaP	EUR /kg P	€ 0.50	Expert judgement
$CaCl_2$	EUR /kg	€ 0.20	Expert judgement
HCl 30%	EUR /ton product	€ 88.00	Vivochem/Brenntag, quote Jan 2023 (VFA STOWA report)

ASSUMTPIONS FOR INVESTMENTS AND MAINTENANCE

Parameter	Unit	Value
Maintenance	%	2%
Depreciation	year	20
Interest	%	4%
Markup factor		1,8

CO_2 EMISSION FACTORS

Parameter	Unit	Value	Source
Electricity	kg/kWh	0.328	Energiemix-NL, jan 2024
NaOH 100%	kg/kg	0.88	EcoInvent
$FeCl_3$	kg/kg	0.86	EcoInvent
$CaCl_2$	kg/kg	0.50	EcoInvent
HCl 30%	kg/kg	0.91	EcoInvent (Wilp project)
Sludge final treatment	kg/kg	0.05	STOWA IPMV
Sludge transport	kg/kg	0.013	STOWA IPMV
Total sludge production	kg/kg	0.063	
NaOCl (100%)	kg/kg	1.00	STOWA IPMV
Citric acid (100%)	kg/kg	1.80	STOWA IPMV
Antiscalants	kg/kg	1.73	STOWA IPMV
Triple superphosphate	kg/kg P	1.76	EcoInvent
GFH granules	kg/kg	2.1	Supplier