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# NuReDrain Webinar II:

# P recovery and P removal modelling

# Practical issues



- Please mute yourself.
- Feel free to ask questions in the chat.
- The webinar will be recorded.
- Handouts will be put available afterwards.

# NuReDrain

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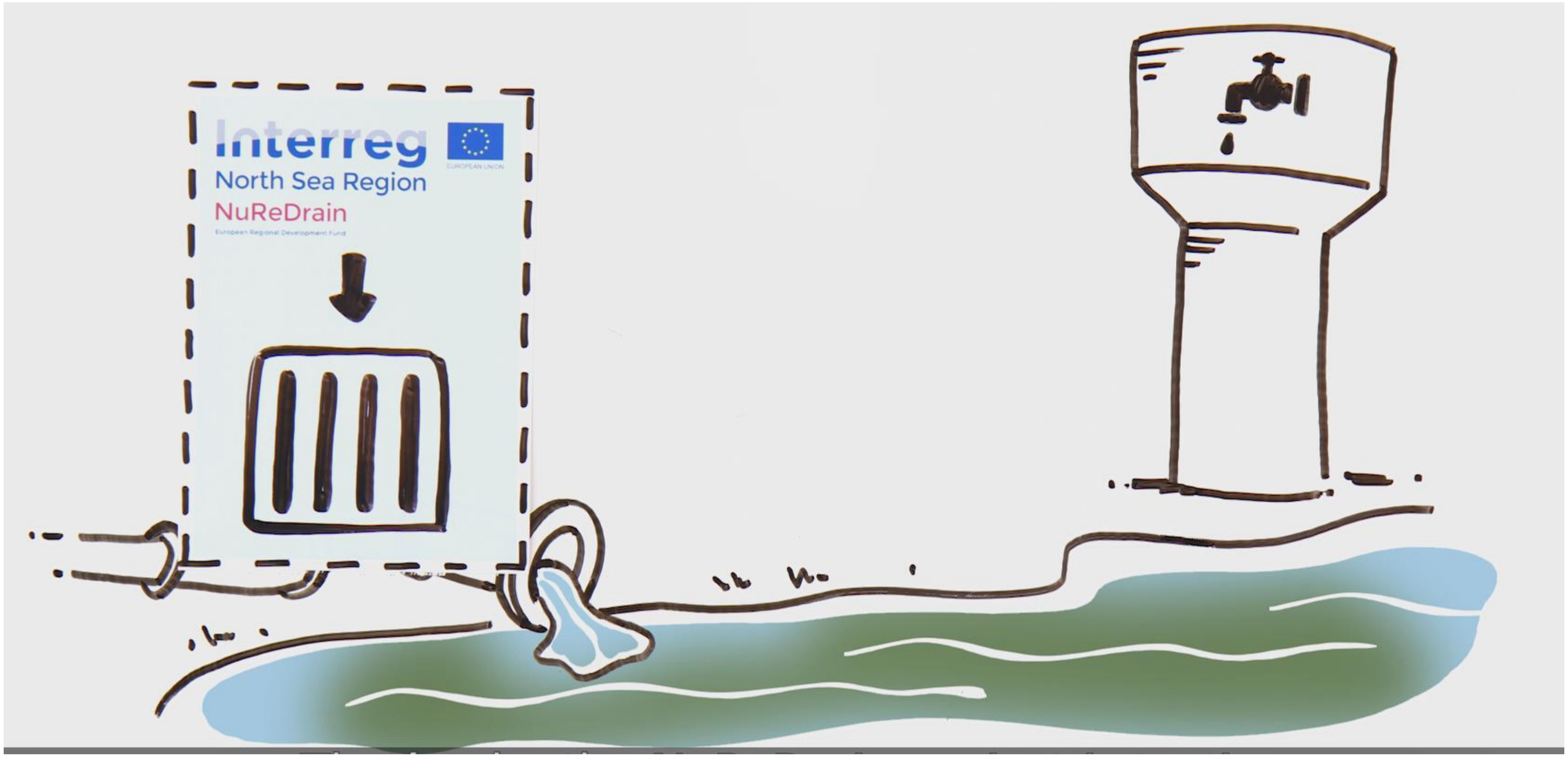
- Nutrient Removal and Recovery from Drainage water
- 1/3/2017 – 30/9/2021
- Interreg North Sea Region
- Project cost: € 2 674 405 - Fund: € 1 337 203
- 11 partners in 3 countries

# Project goal



# Project goals

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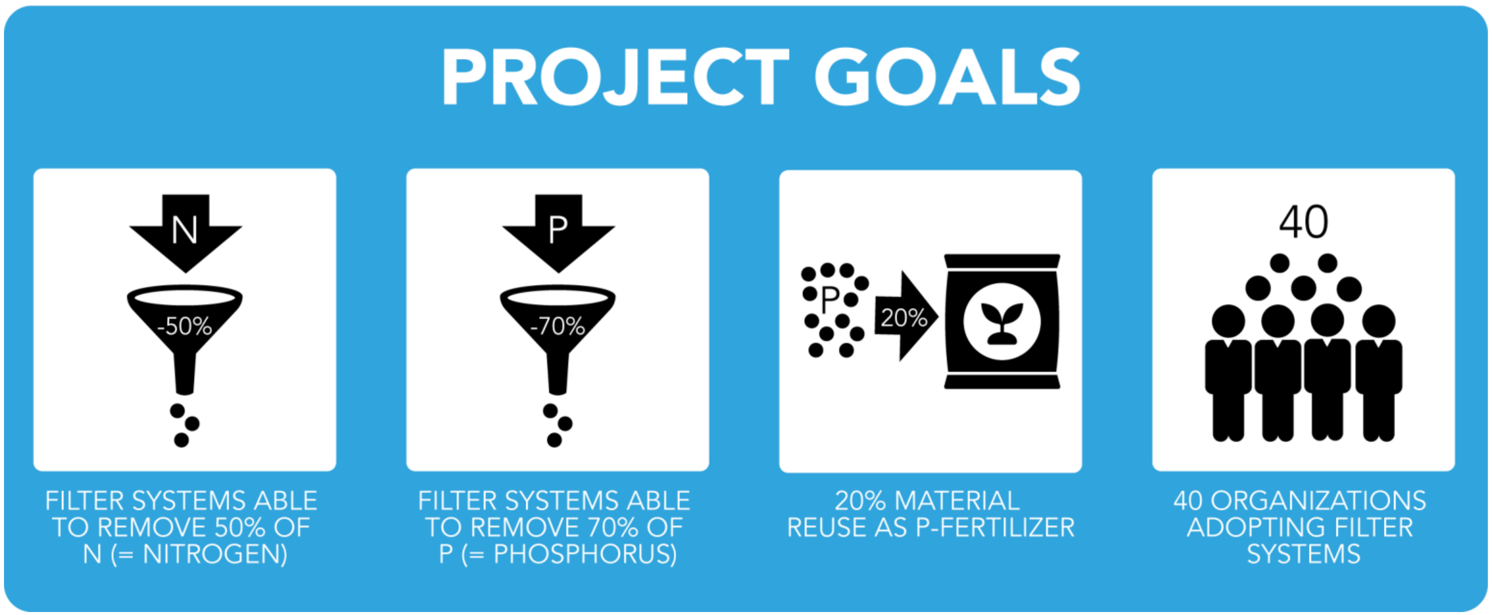

# Agricultural waters

drainage water

greenhouse effluent

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surface water

water reservoir for drinking water production

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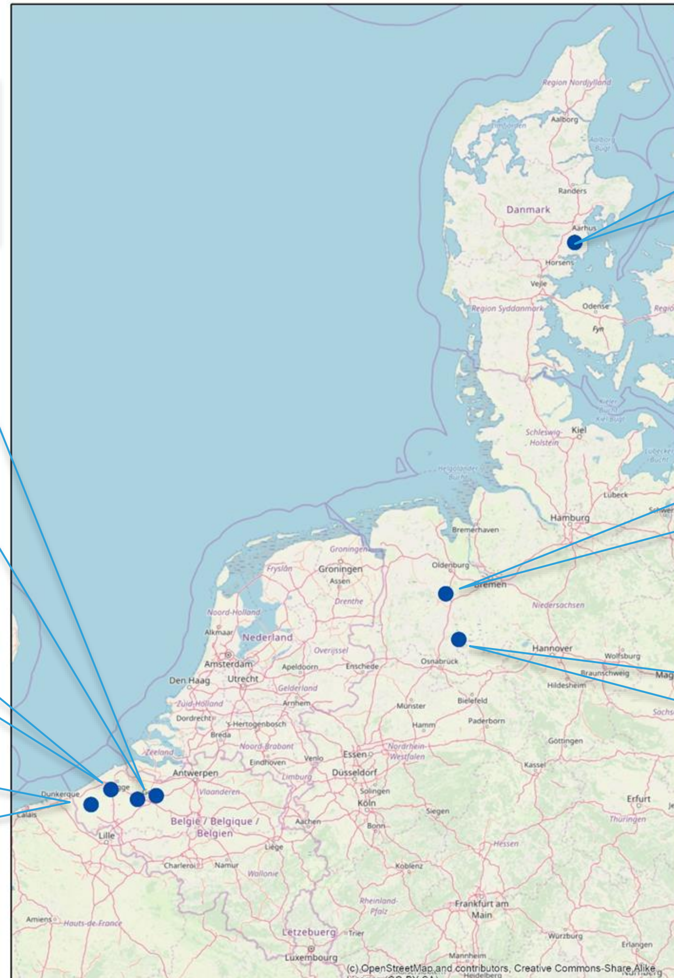
# 6 field cases

Greenhouse effluent
N + P removal

Drainage water
P removal

Water reservoir
P removal




Drainage water
N + P removal

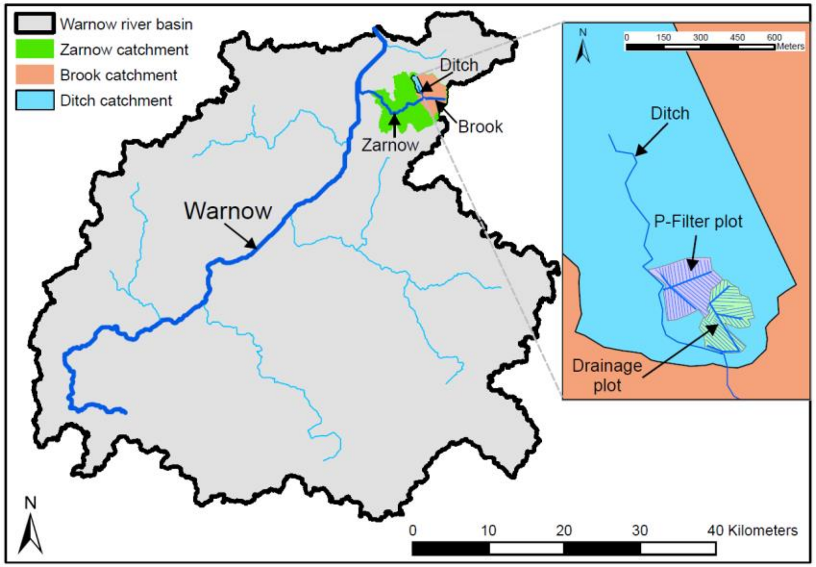
Surface water
N removal

Drainage water
P removal

## P recovery

Integration of P-adsorbing material in a circular process

## Modelling



# Reuse of saturated filter materials as fertilizer for ornamentals and vegetables

***Els Pauwels***

Ornamental Plant Research (PCS), Belgium

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# Problem statement

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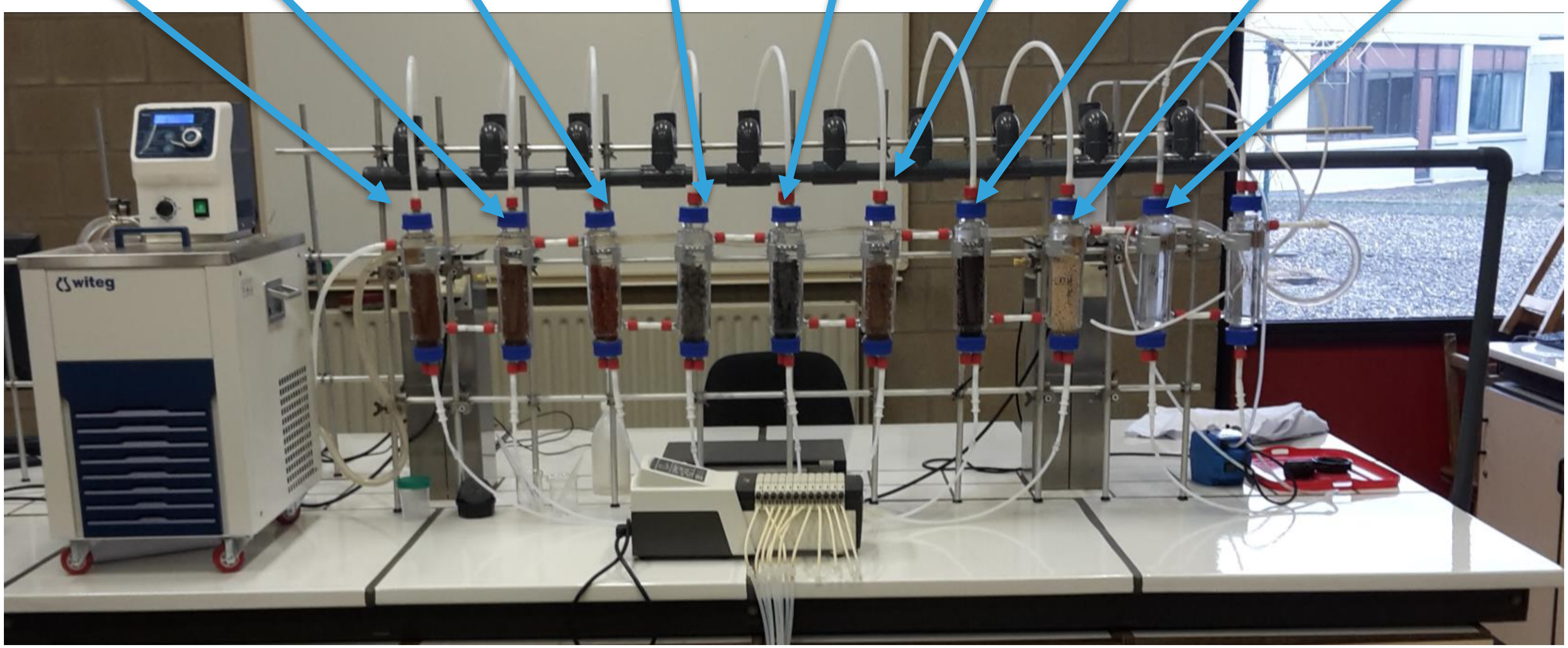
- Phosphorus recovery potential

# P-removal – Column tests

- $PO_4$-P solution: 0.5 ppm P
- Bed height: 14 cm ⇒ corresponds with a bed volume of 150 mL
- Temperature: 20 °C
- Flow rate: 0.66 L/24 h

ICS, Diapure, Redmedite, BaseLith, LiDonit, Vito A, Vito B, LDH, FerroSorp



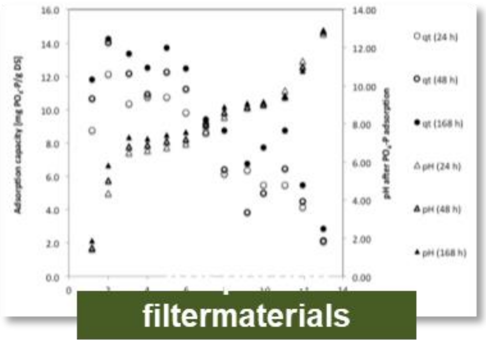
# Problem statement

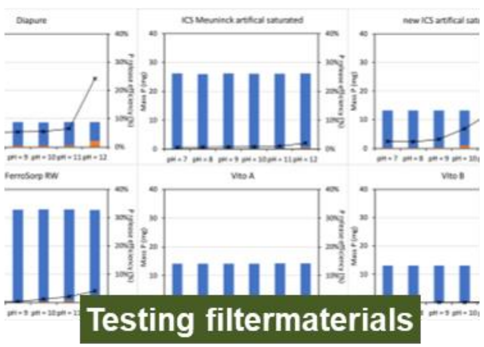
Available: ICS (Iron coated sand) :

- Waste product from drinking water production
- Good removal of P - rich drainage waters
- High conductivity of filters (depending on size of particles)
- (Sufficiently) available and (relatively) cheap

- Reuse as a fertilizer without treatment?

# Trials at PCS


filtermaterials


Testing filtermaterials


Test on azalea

## Pot trial 2017:

- On azalea
- Low pH
- From ICS, there was almost no natural desorption of P, a little desorption of N
- Plants with ICS were of a lower quality compared with the control due to a P shortage

# Trial PCS 2018: Buxus, Lavendula and Hedera

**Table: Overview N and P dose for each tested species**

| | Treatment 1 | | | Treatment 2 | | | Treatments 3 | | |
|---|---|---|---|---|---|---|---|---|---|
| | Standard N and Standard P | | | Standard N without P | | | Standard N without P but with 30% ICS granules | | |
| | N (g/l) | $P_2O_5$ (g/l) | K (g/l) | N (g/l) | $P_2O_5$ (g/l) | K (g/l) | N (g/l) | $P_2O_5$ (g/l) | K (g/l) |
| **Lavendula** | 420 | 245 | 665 | 420 | 0 | 663 | 420 | 0 | 663 |
| **Buxus** | 625 | 315 | 420 | 623 | 0 | 414 | 623 | 0 | 414 |
| **Hedera** | 525 | 315 | 420 | 537 | 0 | 414 | 537 | 0 | 414 |

Growth with standard N and standard P best
No phytotoxicity effects
Difficult to remove ICS grains for analysis

# Trial PCS 2018: Buxus, Lavendula and Hedera

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28/05/2018


13/06/2018


18/06/2018


17/07/2018


30/07/2018


03/08/2018


09/08/2018


13/08/2018


04/09/2018

# PSB

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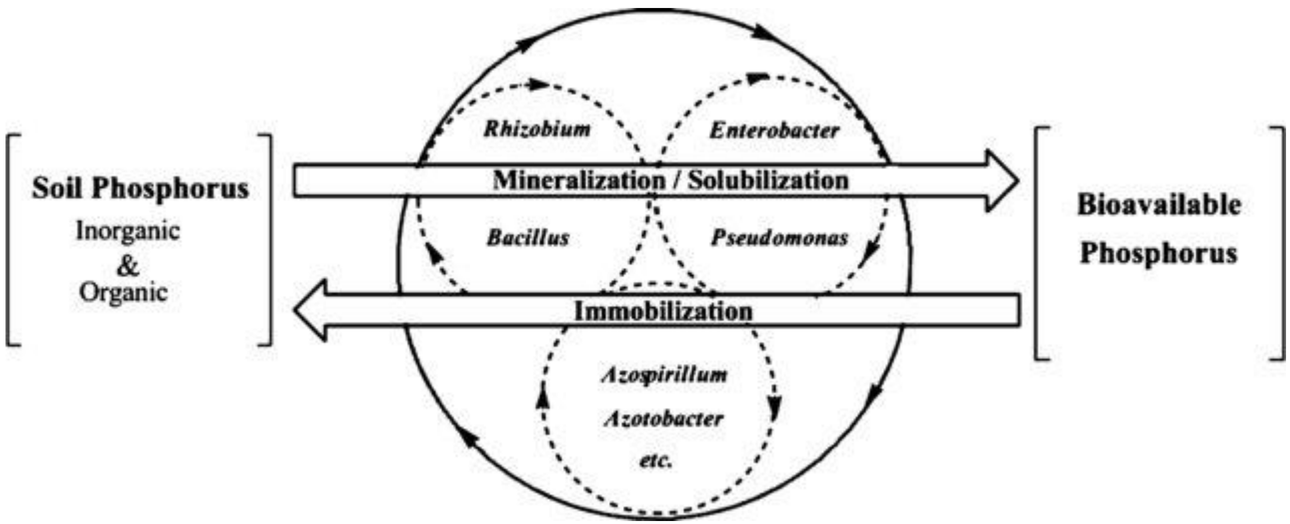
## Schematic diagram of soil phosphorus mineralization, solubilization and immobilization by rhizobacteria




- Predominant bacterial PSB's (sharma et al, 2013):
  - Pseudomonas spp.
  - Bacillus spp.
- P – SOLUBILIZING POTENTIAL depends on :(Sharma et al, 2013)
  - Iron concentration in the soil
  - Soil temperature
  - C and N sources available

# Trial PCS 2019: Hedera

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proefcentrum voor sierteelt

# Trial PCS 2019: Hedera

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| Treatment | |
|---|---|
| 1 | Standard N and Standard P |
| 2 | Standard N without P |
| 3 | Standard N without P but with 30% ICS granules |
| 4 | Standard N without P but with 30% ICS granules + dose 1 of PSM1 |
| 5 | Standard N without P but with 30% ICS granules + dose 2 of PSM1 |
| 6 | Standard N without P but with 30% ICS granules + dose 1 of PSM2 |
| 7 | Standard N without P but with 30% ICS granules + dose 1 of PSM3 |

Potting: End of May
1,5 L pot
Open air

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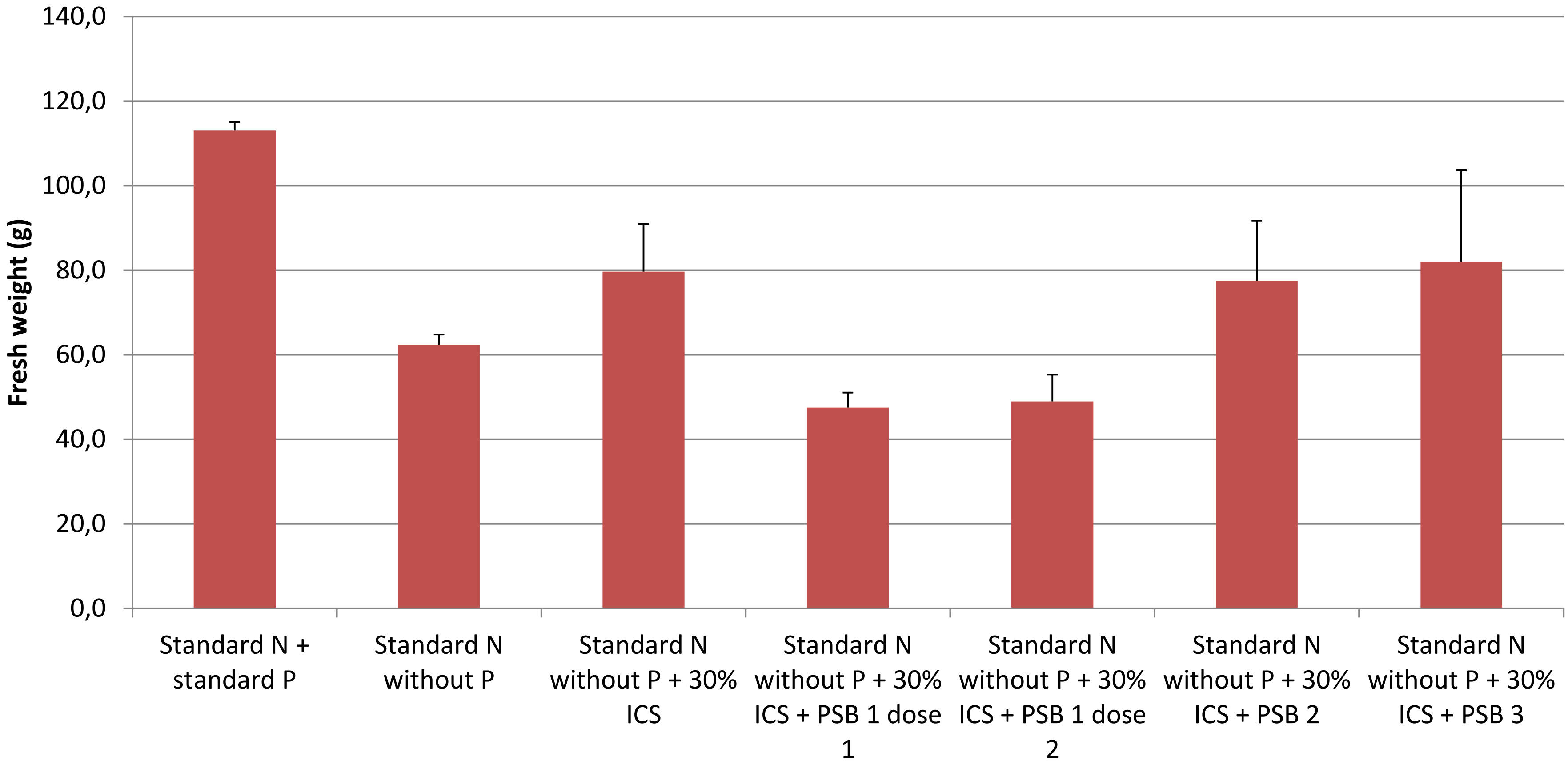
# Trial PCS 2019: Hedera



# Trial PCS 2019: Hedera



# Trials Inagro in agriculture

inagro

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Endive:

- growth chamber experiment + pot experiment
- Use of ICS as a P – fertilizer
- Use of PSB's
- Evaluation of commercial products

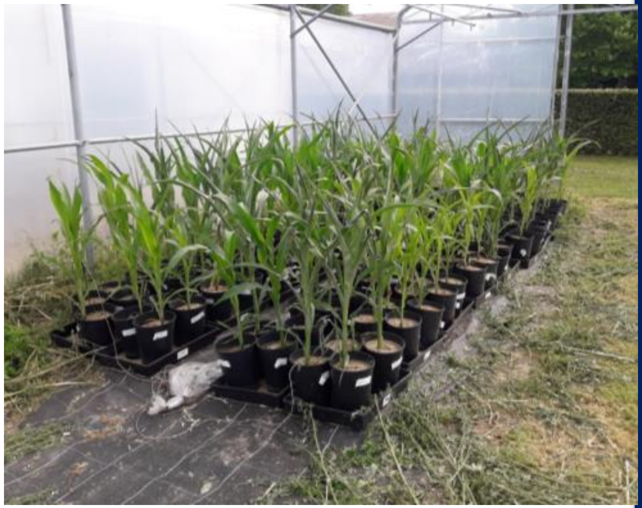
Maize:

- Pot experiment
- Evaluation of commercial products

# Pot trial maize P-fertilisation with ICS

**Phosphorous fertilization value of P-saturated ICS in combination with PSB (P-solubilizing bacteria) in maize**

1. Control (untreated)
2. APP (ammonium polyphosphate) = reference
3. TSP (triple superphosphate)
4. PT mix + ICS
5. PT mix
6. PT mix + TSP
7. Pseudomonas putida + ICS
8. Pseudomonas putida
9. Pseudomonas putida + TSP



# Trials Inagro in agriculture

Overall conclusion pot trials endive and maize

- -> fertilisation treatments with TSP or APP have significant the highest relative yield
- -> no positive effects of the use of PSB's in combination with ICS
- No indication that phosphorus rich material (ICS) has a potential as P-fertilizer
- No added value of PSB's in combination with ICS

# Other possibilities to use ICS?

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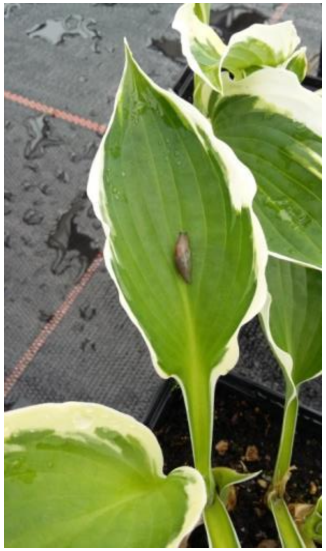
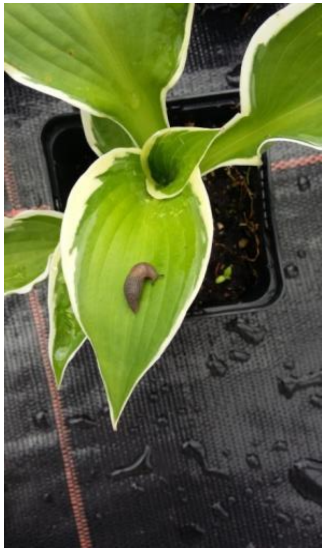
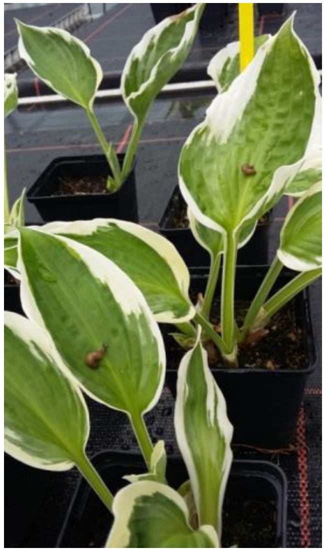
- Against slugs?







- Ironmax Pro (2,4% iron phosphate) (10721P/B),
- Sluxx (3% iron phosphate) (9722P/B),
- Derrex (3% iron phosphate) (9904P/B)

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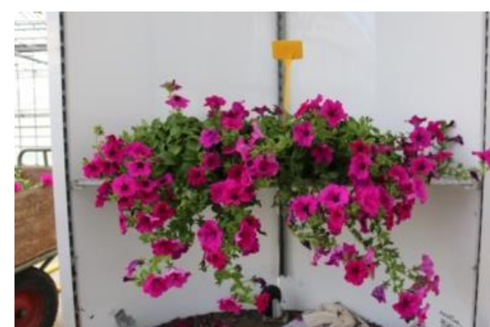
# Trial PCS: 14 different plant species

*Azalea indica* ‘Fluostern’
*Calluna vulgaris* 'Siska'
*Camelia*
*Chamaecyparis lawsoniana* Elwoodii
*Chrysanthemum* ‘Salomon Surfer mauve and *Chrysanthemum* Sevilla orange bicolor "Josevor"
*Erica x darleyensis* 'kramer's rood'
*Euonymus fortunei* 'Emerald Gaiety'
*Hydrangea paniculata* 'Phantom'
*Lavendula angustifolia* 'Munstead’
*Pelargonium zonale* Dark ‘Clara White’
*Petunia surfinia* var. Purple
*Rhododendron ponticium* 'Graziella'
*Thuja occidentalis* 'Brabant'
*Waldsteinia ternata*

# Trial PCS: 14 different plant species





# Trial PCS: As addition to the substrate? Chlorophytum



- Evaluation at end of trial (16/07/2018)

rooting 5 (left) – rooting 7 (right)

| | # rootings trough pot | rootscore 1-7 | Fresh weight (13 plants) | Visual plant quality |
|---|---|---|---|---|
| With ICS | 8,3 | 6,2 | 333,13 | 9 |
| Without ICS | 8,5 | 6,2 | 310,37 | 9 |

# Other possibilities to use ICS?

## Trial PCS: As addition to the substrate?

- Evaluation at end of trial

**Chlorophytum**

left without ICS – right with ICS

# Exceptions

- **Chrysanthemum**

- **Chlorophytum**

left without ICS – right with ICS

- **Petunia**

# Trial PCS 2020: ongoing

20 plants/treatment

- 1. Control
- 2. 30% ICS grains
- 3. 30% pellets

# Trial 2020



30 %

# Other possibilities to use ICS?

| | | |
|---|---|---|
|  |  |  |
| Standard N + standard P | Standard N without P | Standard N without P + 30% ICS |

# Other possibilities to use ICS?

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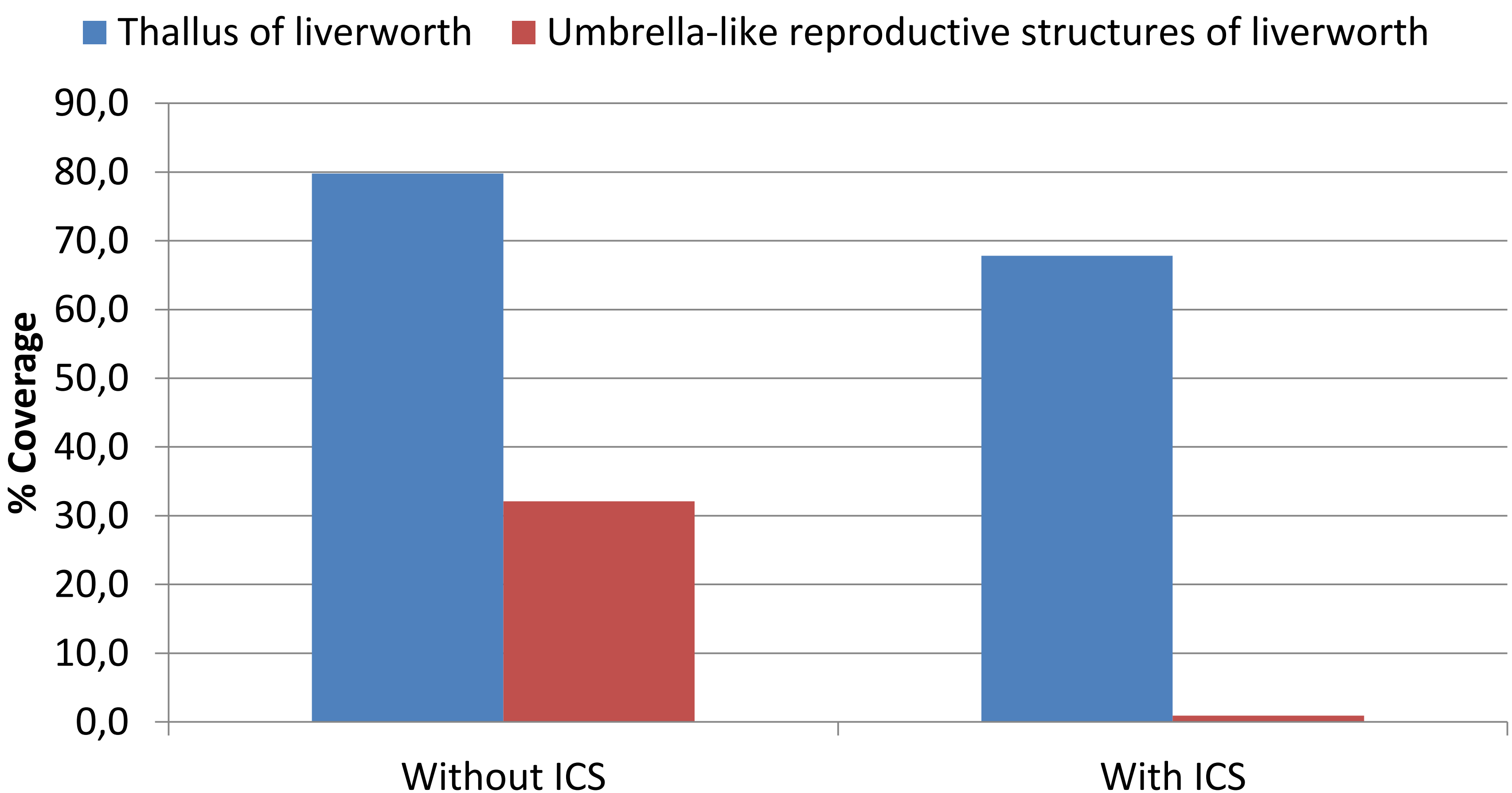
# Other possibilities to use ICS?

*Camellia*



# Other possibilities to use ICS?

- As a cover material?

# Other possibilities to use ICS?

# Other possibilities to use ICS?

# Q & A

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- http://northsearegion.eu/nuredrain/
- Subscribe to our newsletter: https://northsearegion.eu/nuredrain/news/
- Els Pauwels- els.pauwels@pcsierteelt.be -+32 9 353 94 88

# Webinar II: Recovery of phosphorus by chemical treatment

Nico Lambert – KU Leuven
Process & Environmental Technology Lab
KU Leuven

# Introduction



- Water flows from agriculture, e.g.,
  - Drainage water originating from tile drained agricultural fields
  - Greenhouse effluent

→ contain phosphate amounts of unused fertilizers
→ above the standard limits for surface water

**Proposed solution**:
Adsorption technology using Al and Fe based P-adsorbing materials: Iron Coated Sand (ICS), Vito A and B, DiaPure.

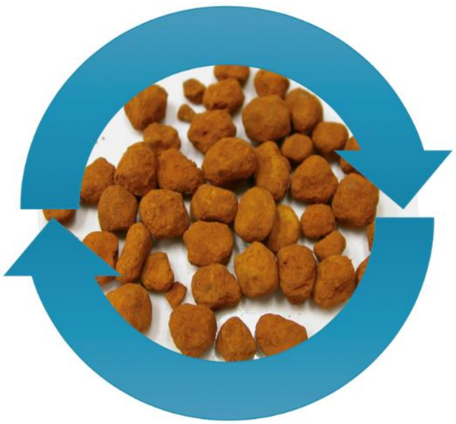
**Relevant research question:**
What about the saturated adsorption material: should it simply be disposed of as solid waste? When is recovery/regeneration recommended?

Integration of P-adsorbing material in a circular process

# Introduction

**Prospects for P-recovery:**

- The main objectives:
  - **Regeneration of the saturated sorbents** making it reusable in several adsorption/desorption cylces and
  - **Recovery of phosphorus** by precipitation or used directly with irrigation water as fertilizer .
- The reusability of the granules is as important (or even more) than recovering phosphate
- Different desorption reagents: inorganic and organic acids, chelating agents and alkaline solutions, are already proposed in the literature
- A desorption process using an **alkaline** solution is proposed without harming the adsorbing material.



Integration of P-adsorbing material in a circular process

# Introduction

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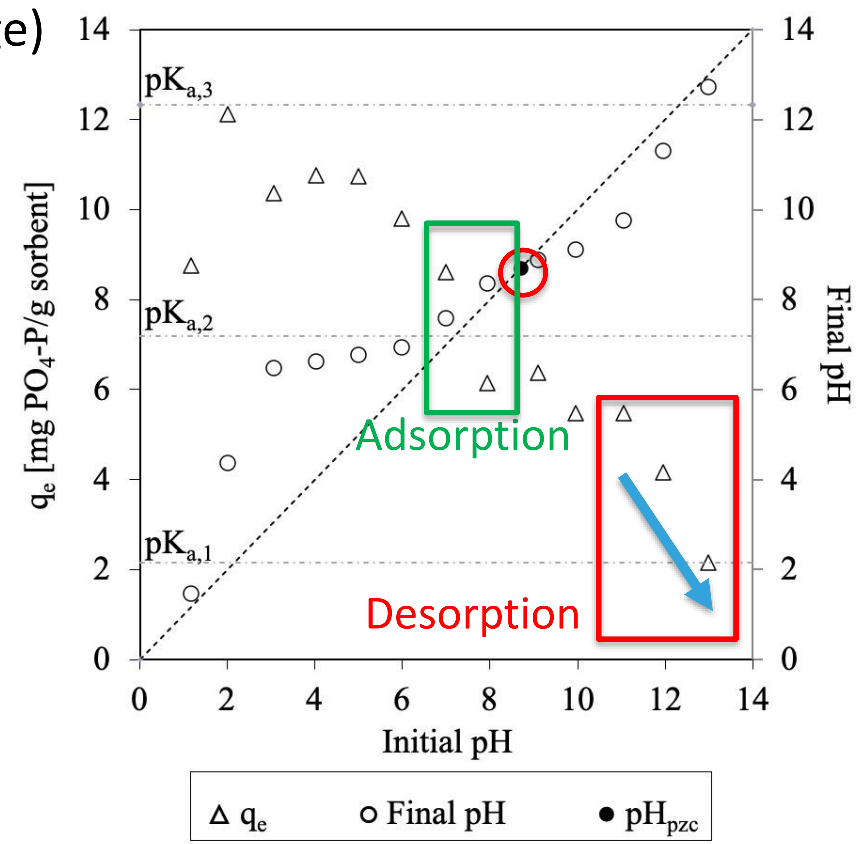
**Theoretical basis:**

- The influence of initial pH on the adsorption capacity $q_e$ for ICS
- Adsorption/desorption are **balancing processes** until an equilibrium is reached!
- pH 8.7 = $pH_{PZC}$ (Point of Zero Charge) = final pH is equal to the initial pH
- pH range 1 - 8.7: high $q_e$
- pH range 8.7 – 13: low $q_e$
- pH>11 the $q_e$ drops considerably

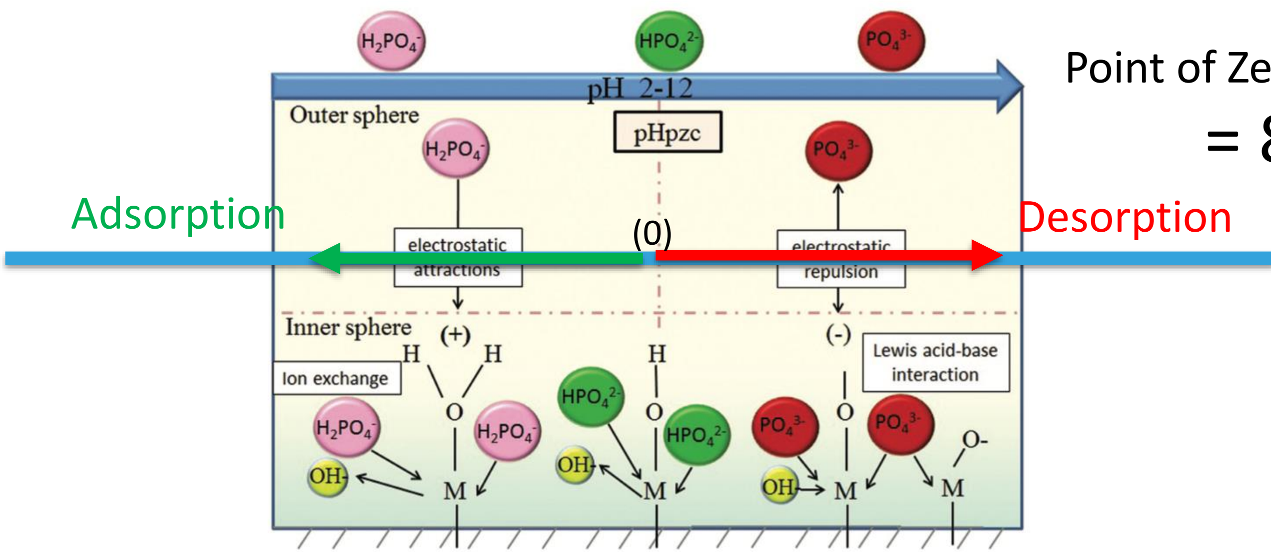


# Introduction



## Theoretical basis:

- Li et al. (2016): higher pH = the phosphate adsorption is affected by
  - the electrostatic repulsion (surface is negatively charged) and
  - increasing competitive effect of OH− ions for the active sites on the sorbent
  - =decreased adsorption capacity.




Point of Zero Charge

= 8.7

Li, M., Liu, J., Xu, Y., Qian, G., 2016. Phosphate adsorption on metal oxides and metal hydroxides: A comparative review. Environ. Rev. 24, 319–332.

# Concept of ad/desorption

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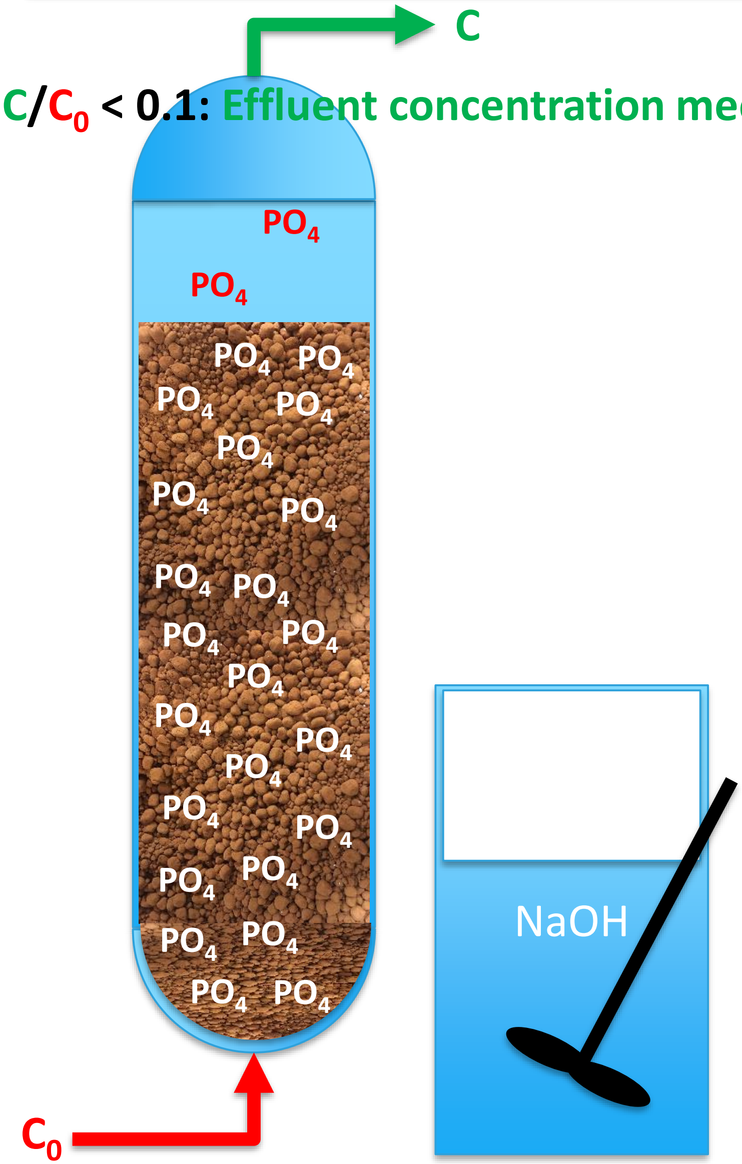
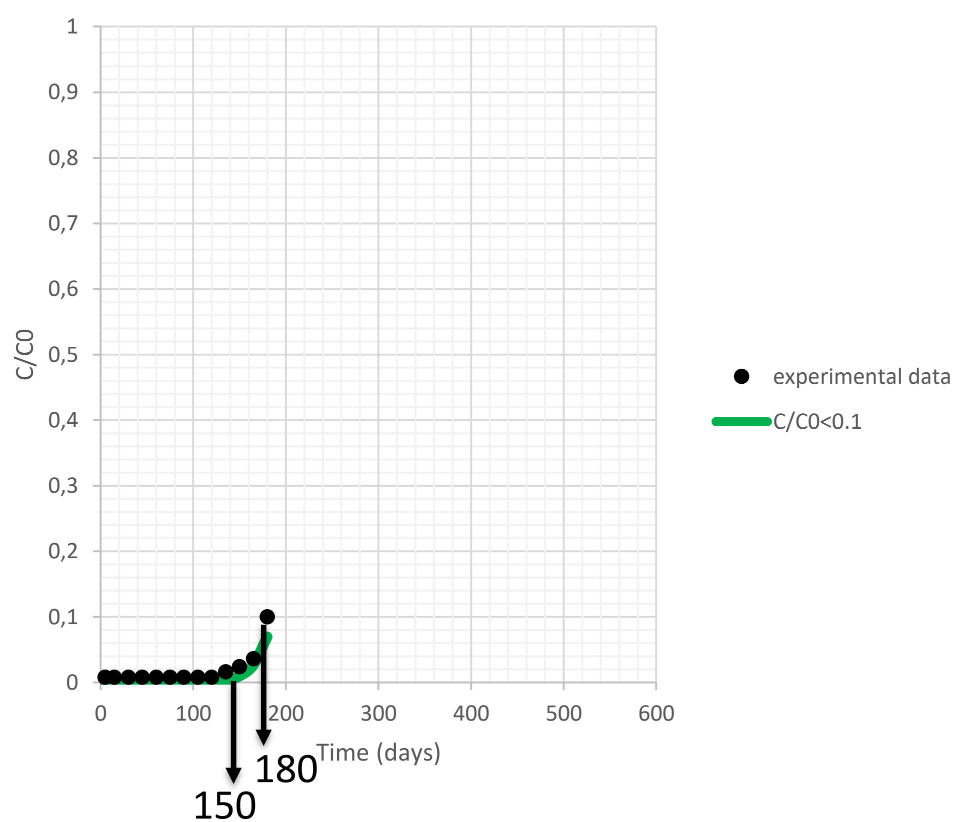
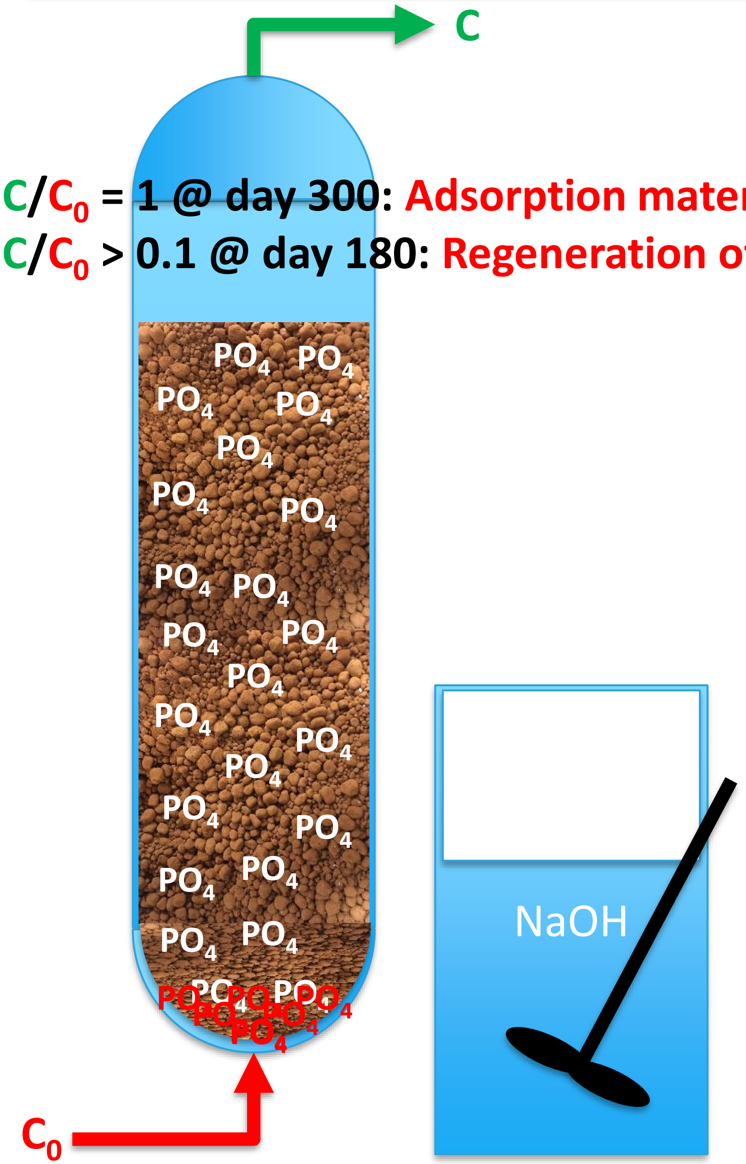
$C/C_0 < 0.1$: Effluent concentration meets the discharge limit




## Adsorption Phase: Day 0-180



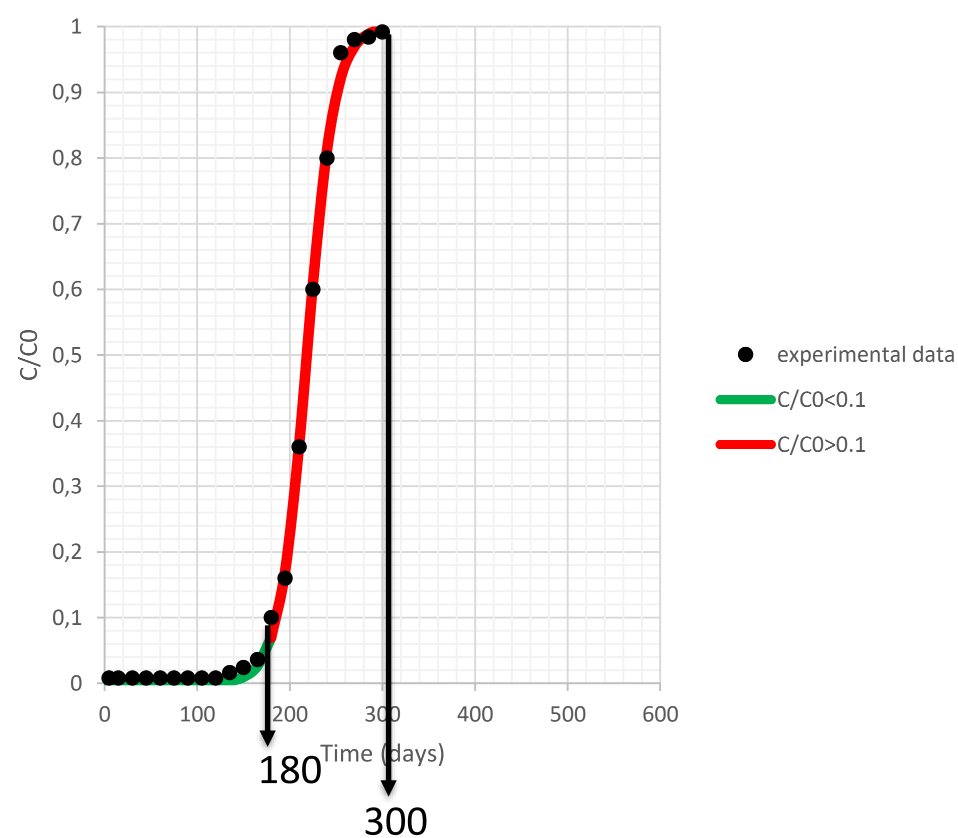
# Concept of ad/desorption




$C/C_0 = 1$ @ day 300: **Adsorption material (ICS) is completely saturated**

$C/C_0 > 0.1$ @ day 180: **Regeneration of ICS is needed**

## Adsorption Phase: Day 180-300



# Concept of ad/desorption

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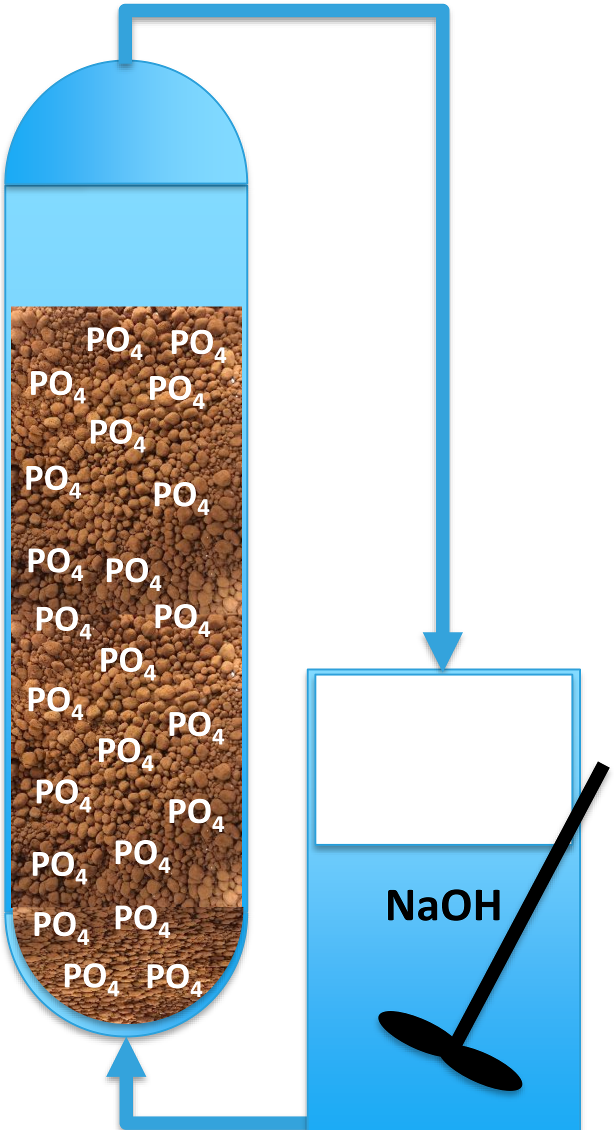
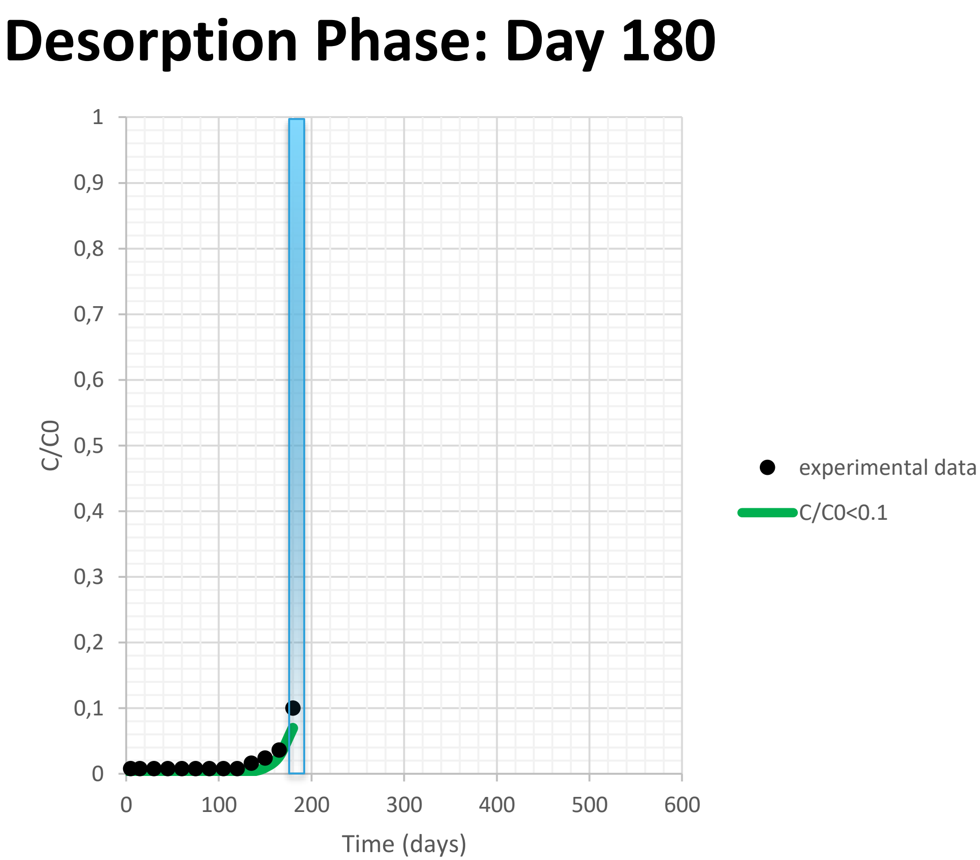
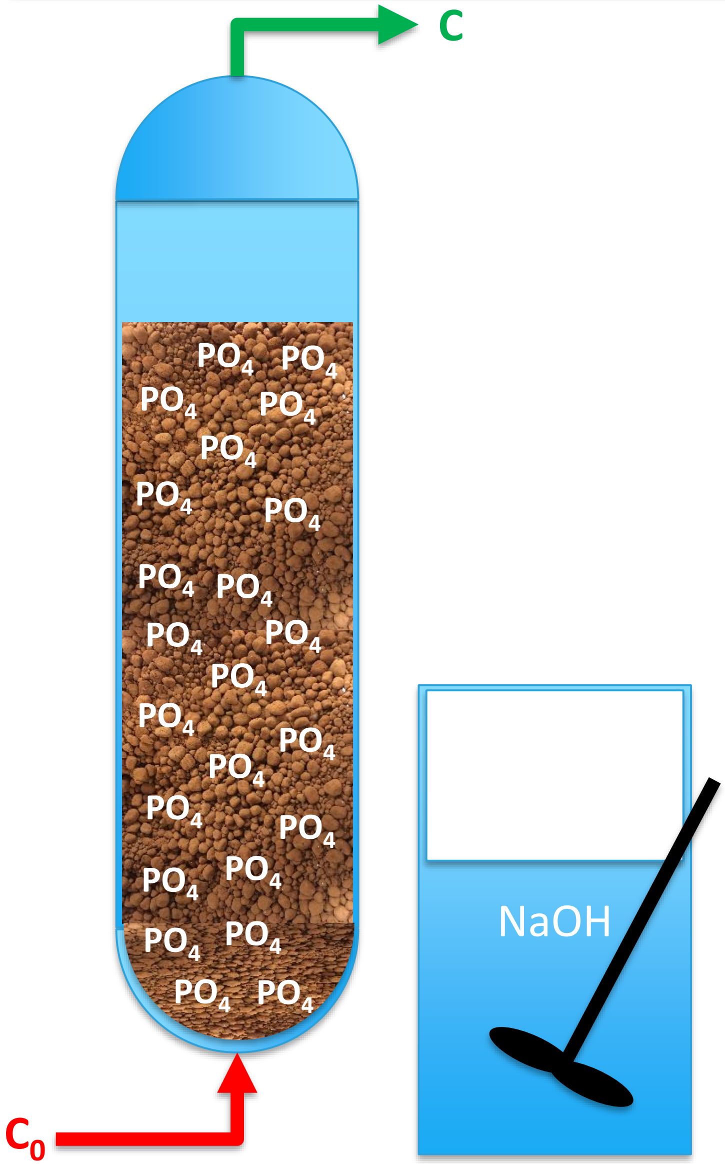


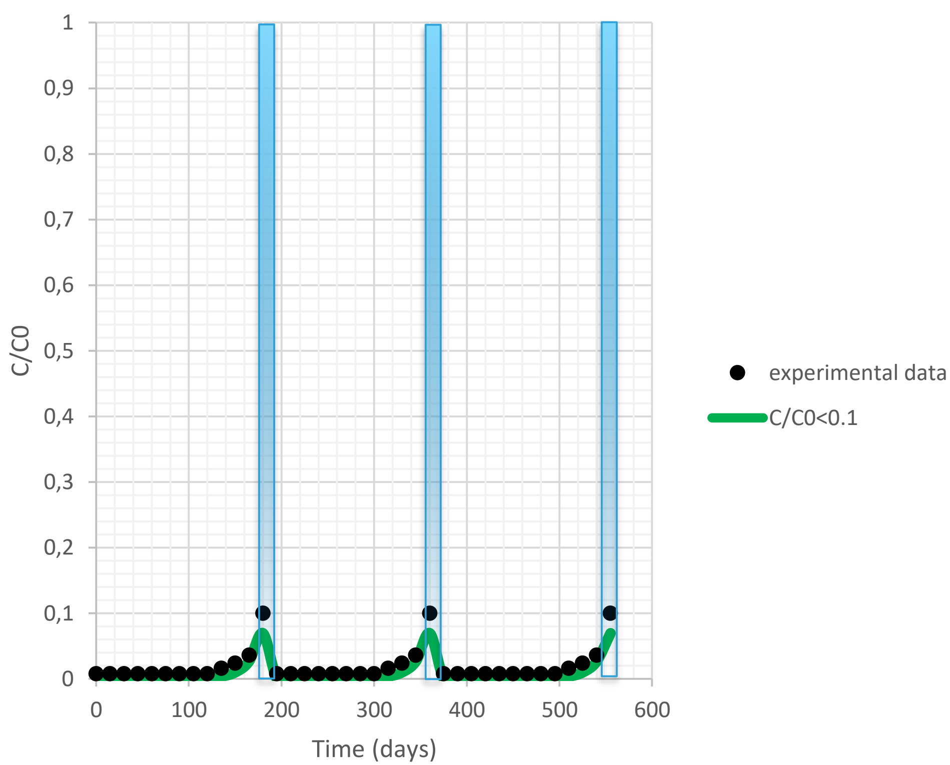
# Concept of ad/desorption






## Regeneration of the saturated sorbent and recovery of phosphorus

### Intermittent regeneration of ICS



# Materials & Methods

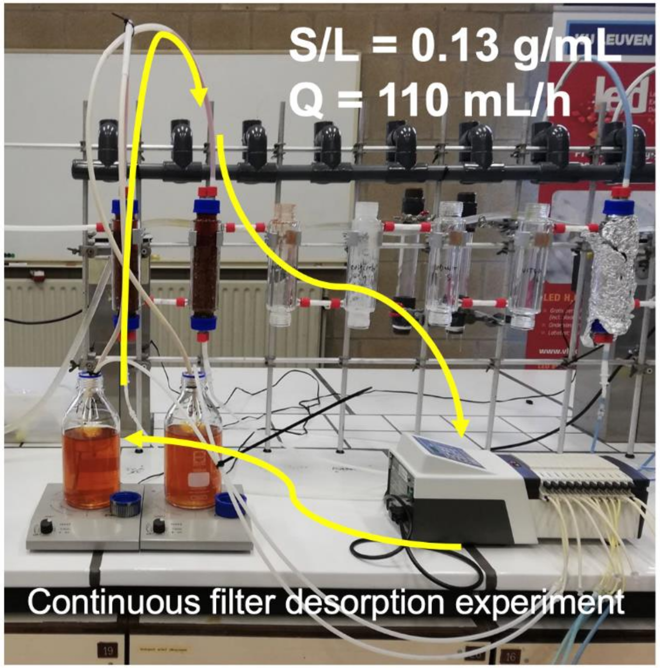
1. **Batch desorption experiments:** 5g of pre-dried saturated ICS was brought into contact with NaOH solution.

   Variable parameters:

   - NaOH concentration (1-0.5-0.1- 0.01- 0.001M),
   - Desorption time (5min-48h)
   - Solid/liquid ratio (S/L= 0.03-1 g/mL)

2. **Continuous filter desorption experiment:** 1 liter of NaOH solution was recirculated over an adsorption column filled with 128 g of saturated ICS granules.

3. **Analysis of the samples: Liquids**: $PO_4$-P determination by ion chromatography after .45 µm filtration. **Solid grains**: SEM-EDX



# Results & Discussion
# Batch experiments

- The composition of 1 g of saturated ICS granules was determined by a complete destruction of the granules by Aqua Regia and ICP analysis:
  - Phosphorus: 15.30 +/-1.25 mg P/g DS **=1.5%P**
  - Iron: 590.7 +/-8.7 mg Fe/g DS **=59%Fe**
- **Figure 1**: A minimum desorption time of 24 hours and a NaOH concentration of 0.1 - 1M is necessary to ensure a sufficiently high desorption efficiency.
- **Figure 2**: The solid over liquid ratio (S/L expressed in g/mL) has a pronounced effect on desorption efficiency. An S/L lower than 0.10 g/mL is recommended.

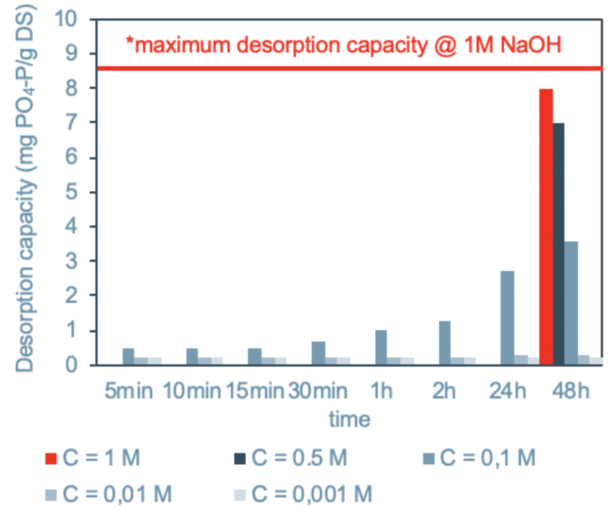



Figure 1: Influence of NaOH concentration and desorption time.

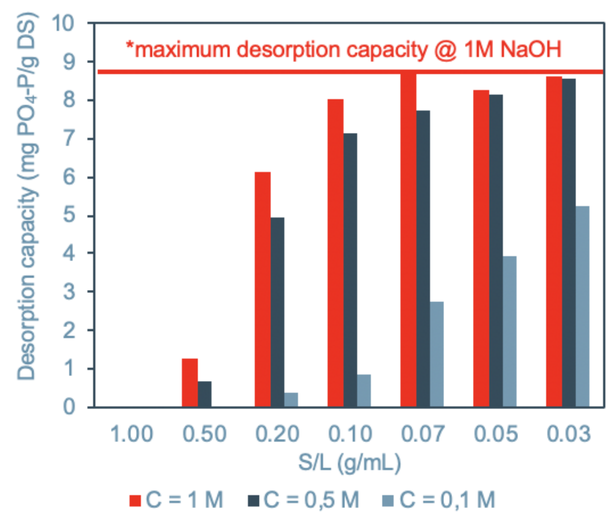



Figure 2: Influence of solid/liquid ratio.

# Results & Discussion
# Continious filter experiments

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- **Figure 3:** Continuous desorption filter experiments show that only a concentration of 0.5 and 1M NaOH lead to a desired desorption of phosphorus from the ICS granule. At least 24 hours desorption time must be provided.

- **Figure 4:** During the first hour of the continuous desorption experiment only 0.4 mg P/g DS and 0.9 mg P/g DS can be leached for a NaOH concentration of 0.5 and 1M respectively. A concentration of 0.1M NaOH desorbed almost no phosphorus.

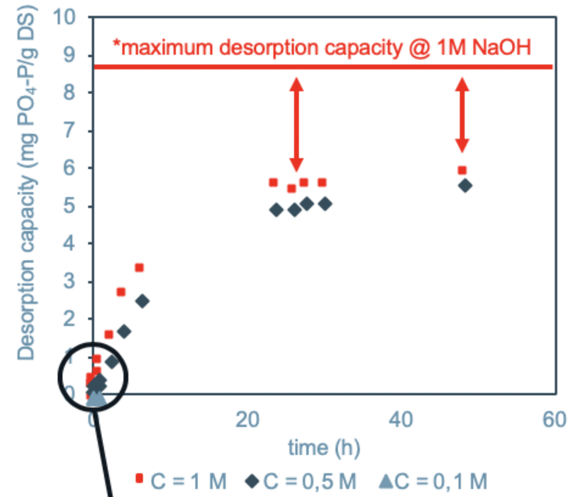



Figure 3: Continuous filter desorption experiment and the effect of the NaOH concentration on desorption capacity

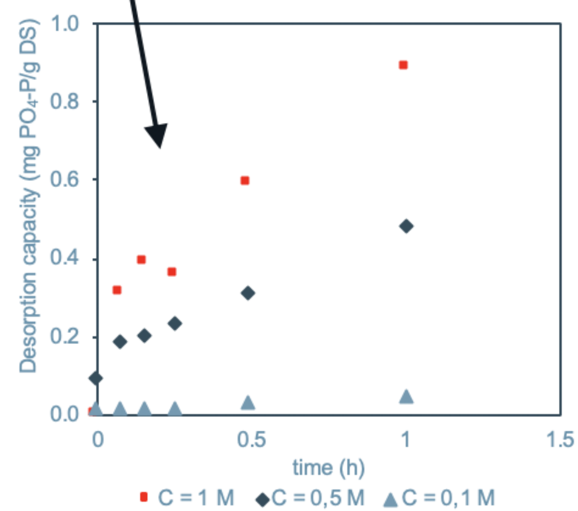



Figure 4: The progress of the desorption during the first hour of the continuous filter desorption experiment

# Results & Discussion
# SEM-EDX analysis

- Energy-dispersive X-ray (EDX) Analysis with a Scanning Electron Microscope (SEM) of saturated ICS from two column experiments.

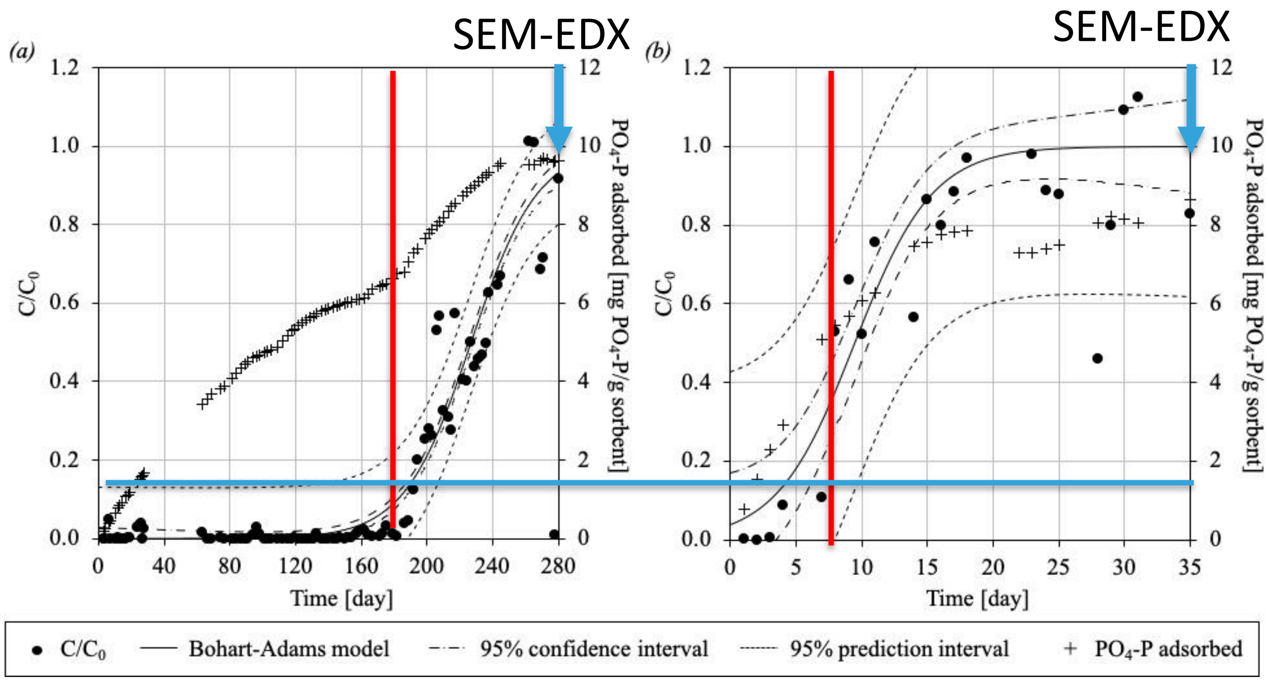



Figure 5: Adsorption column experiments on lab-scale (influent P concentration = 25 mg $PO_4$-P/L) with EBCT= 5.5 h (a) and EBCT= 0.5 h (b)

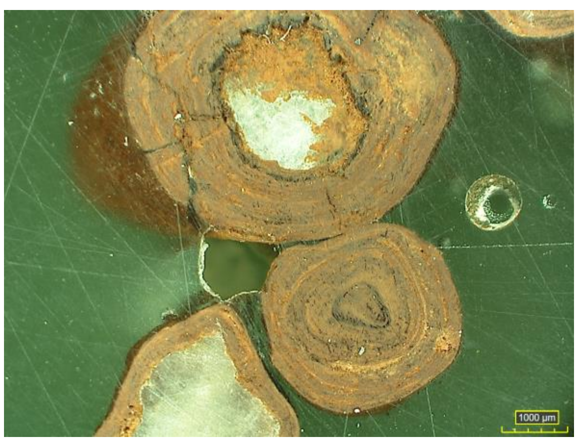
- **Figure 5:** The breakthrough curve of column experiments with an Empty Bed Contact Time (EBCT) of **5.5 h** and **0.5 h** results in a breakthrough time of **180 days and 7 days** respectively.

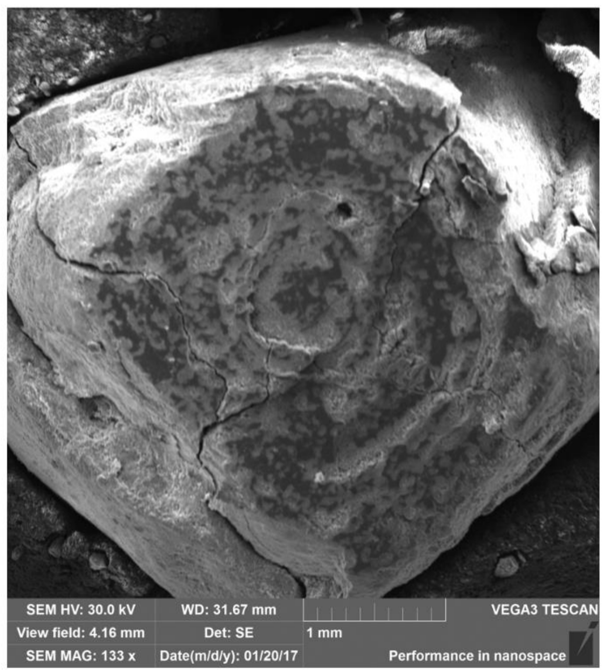
# Results & Discussion
## SEM-EDX analysis

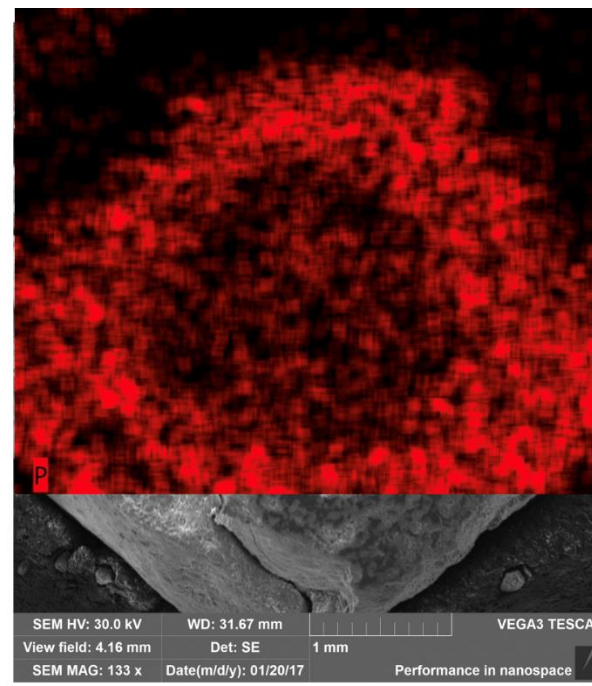
- Energy-dispersive X-ray (EDX) Analysis with a Scanning Electron Microscope (SEM) of saturated ICS from two column experiments.

- **Figure 6:** SEM-EDX of saturated ICS of column experiment with **EBCT of 0.5 h**. The phosphate is mainly adsorbed at the outer layers of granules.

**polished ICS granules embedded in a resin**



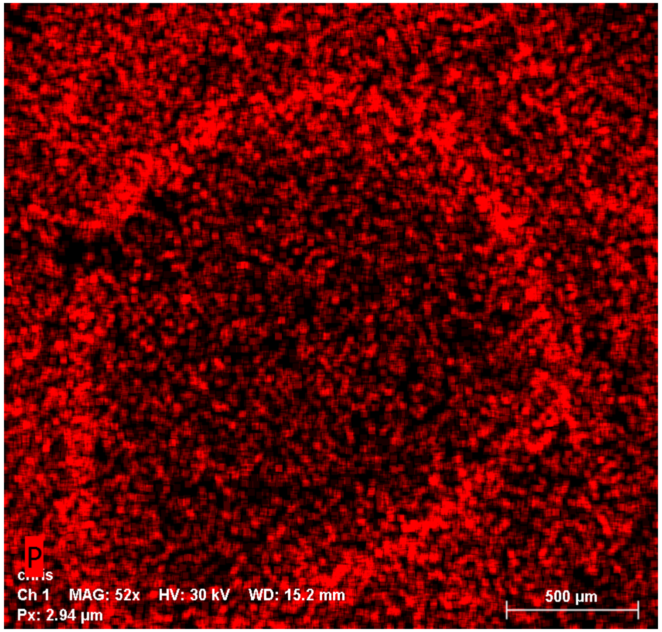



# Results & Discussion
# SEM-EDX analysis

- Energy-dispersive X-ray (EDX) Analysis with a Scanning Electron Microscope (SEM) of saturated ICS from two column experiments.

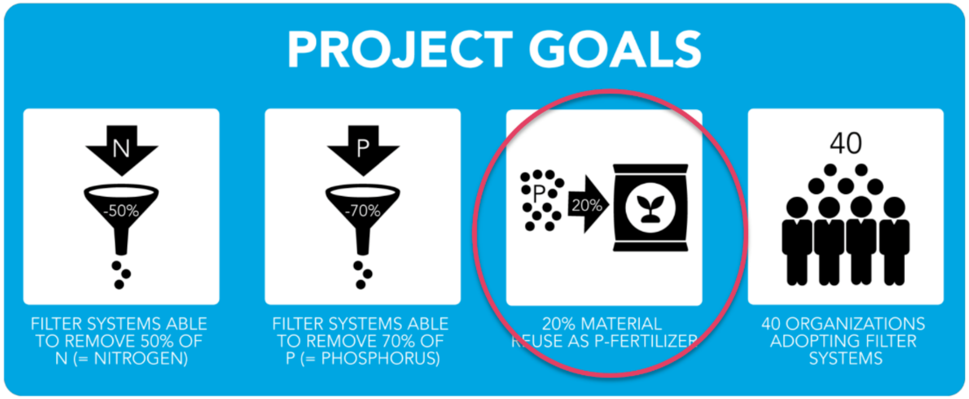
- **Figure 7:** SEM-EDX of saturated ICS of column experiment with **EBCT of 5.5 h**. phosphorous is accumulated at the sand core of the granule = phosphorous migrates towards the core of the granule.

**Si – Fe – P analysis by EDX**



# Conclusions

- Optimal NaOH concentration = 0.5 M
- Optimal contact time = 24 hours or more
- Optimal S/L ratio = 0.10 - 0.05 g/mL
- P-desorption efficiency = 40% @ 0.5 and 1 M NaOH
- Leaching of Fe during the desorption process is a problem
- Desorption of P from the inner layers of the granule will be a problem



# Future perspectives

- **What to do next?**
  - Investigating whether other adsorption materials are better suited for desorption: Vito materials and DiaPure?
  - Looking for ways to reduce desorption pH.
  - Carrying out continuous long-term column tests in which cycles of adsorption and desorption are completed → To do in the coming months.

# Q & A

# **Phosphorus Removal Modelling** – From a Single Filter to an Entire Catchment

**Stefan Koch**, Andreas Bauwe, Bernd Lennartz

Interreg
North Sea Region
NuReDrain
European Regional Development Fund
EUROPEAN UNION

# INTRODUCTION

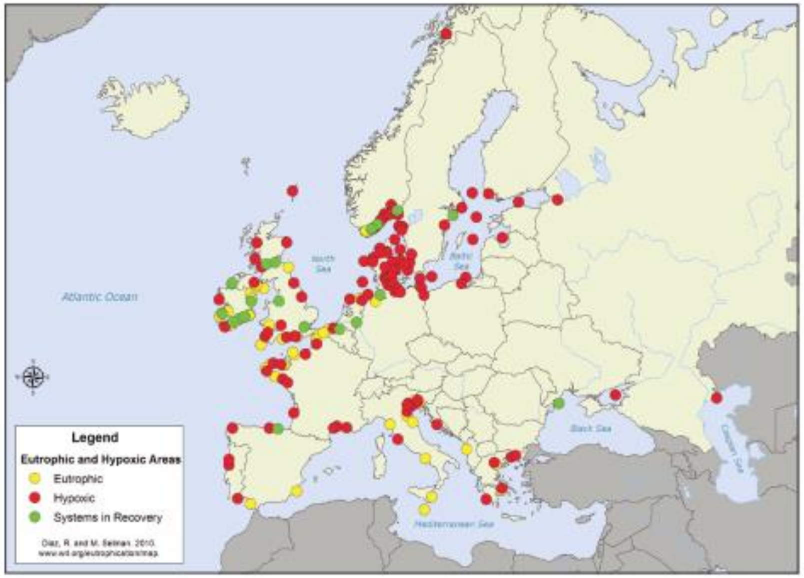
# Introduction

- Eutrophication is a major threat to coastal ecosystems
- Harmful algae blooms may cause deoxigenation of water bodies
    - May not only occur in deep waters of oceans
- Nitrogen (N) and phosphorus (P) inputs from agriculture are a critical source of excess nutrients in surface waters

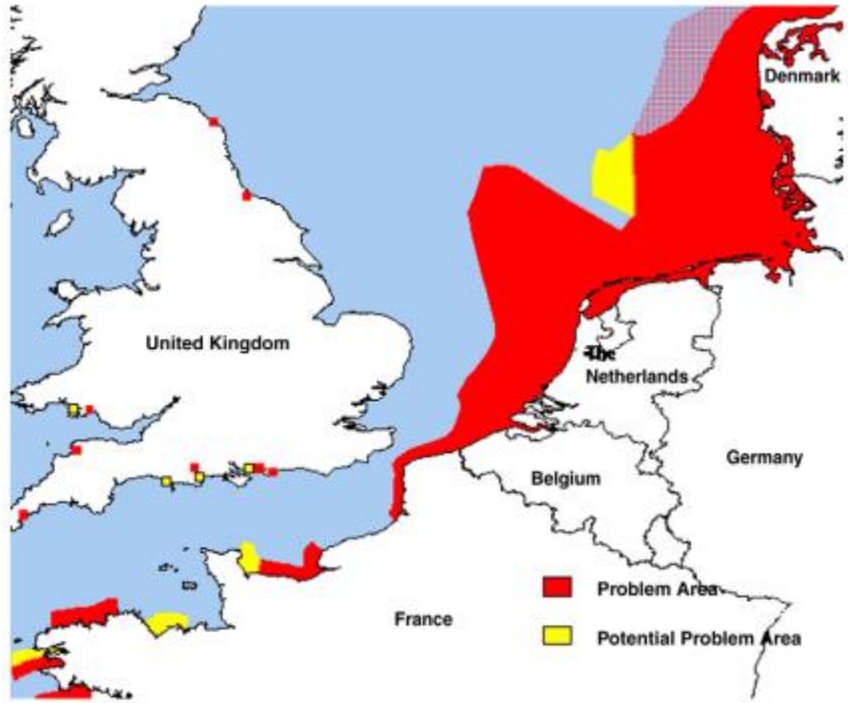



(ESA/ESA/The Guardian/GreatLakesNow)

# Eutrophic Areas in the North Sea




IUCN (2019)




Lenhart et al. (2010)

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# STUDY SITE AND METHODS

# The Kemmelbeek Watershed

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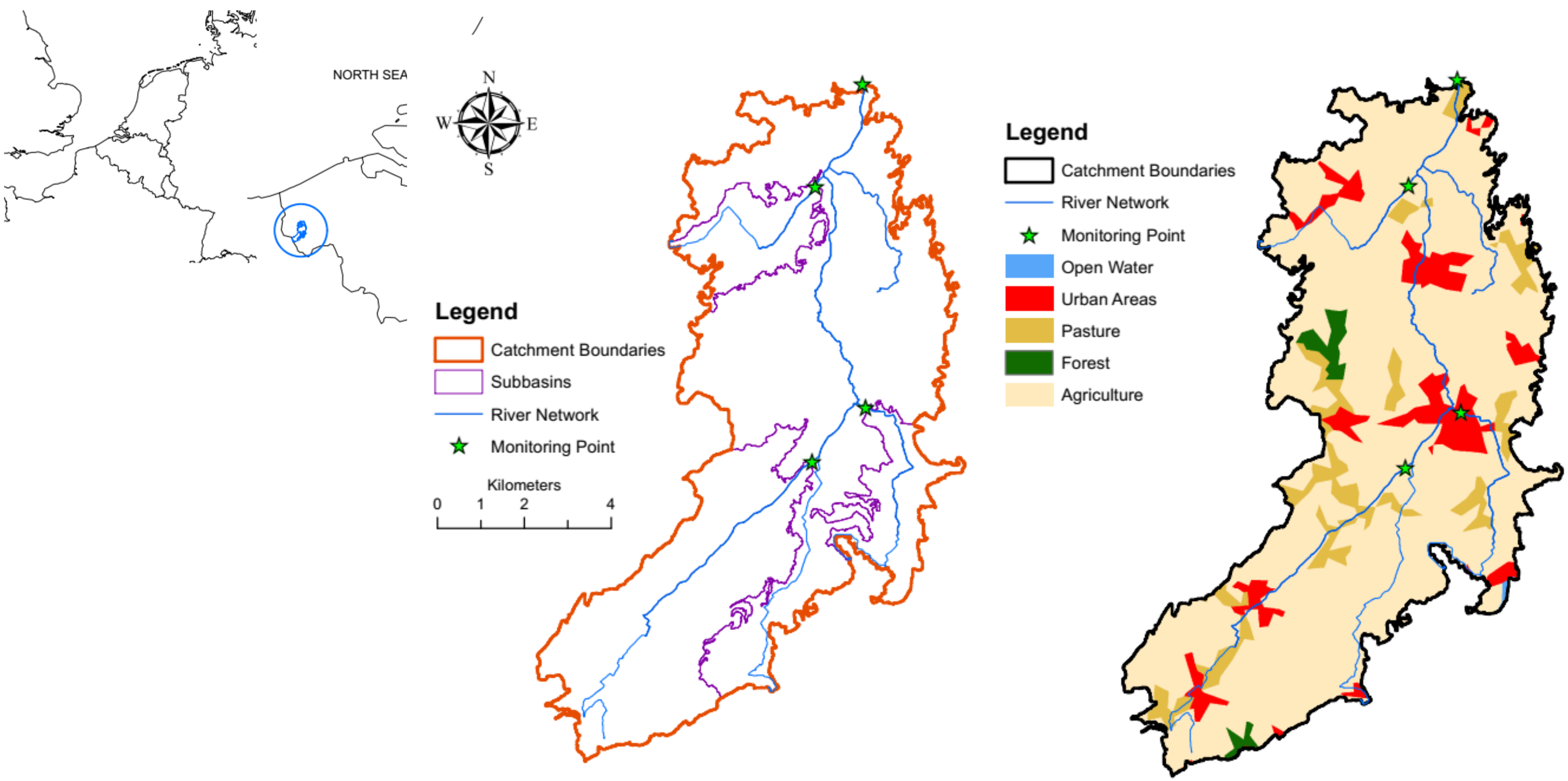
- Belgian (Flemish) Watershed, 74 km², situated in Western Belgium
- Agriculture (61.85 km²; 83%) is the major land use in the Kemmelbeek Watershed



# The Kemmelbeek Watershed

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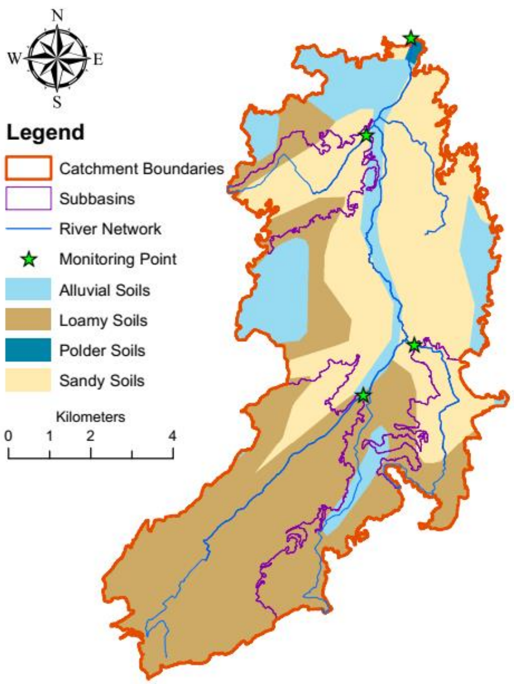
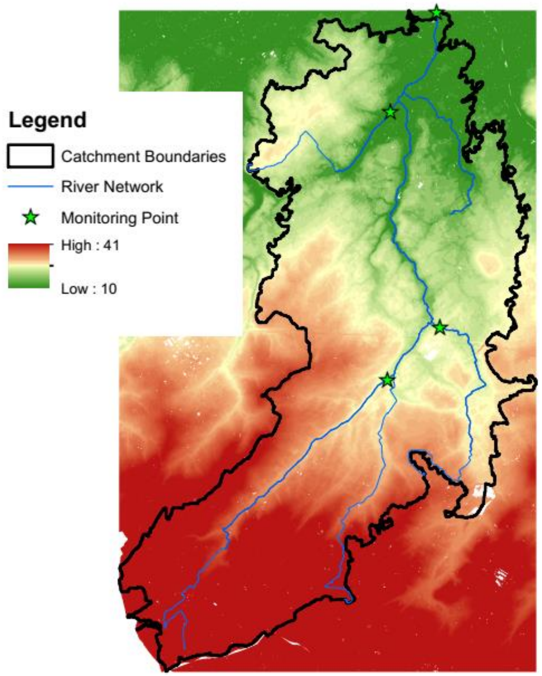
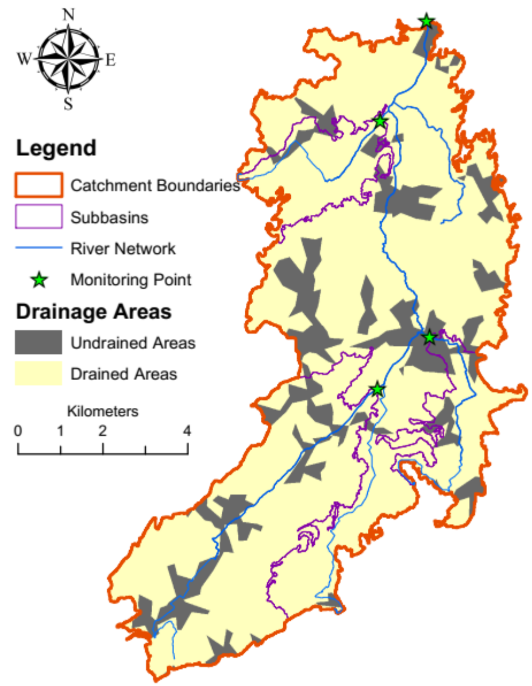
- A heavily tile-drained lowland watershed dominated by loamy soils



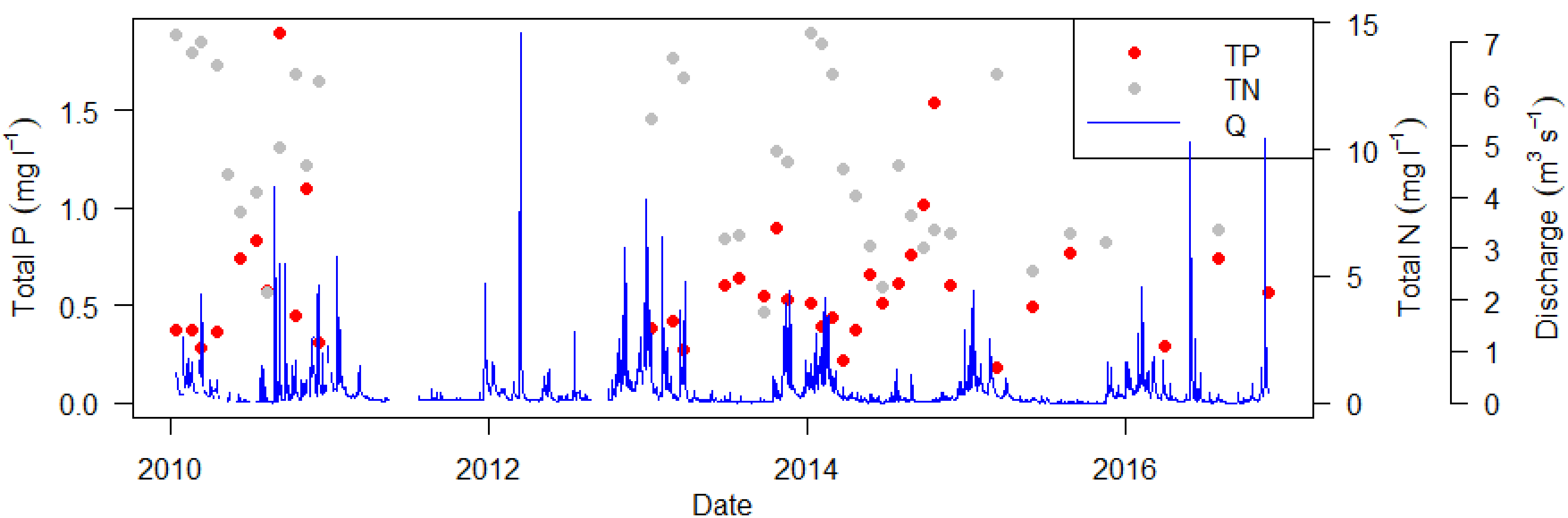



# The Kemmelbeek Watershed

- Elevated TP concentrations in the Kemmelbeek Watershed -> **reduction required**

| | mean | max | min | Mean load yr$^{-1}$ |
|---|---|---|---|---|
| **N (mg/l)** | 9.5 | 14.6 | 3.6 | 8.9 kg ha$^{-1}$ |
| **P (mg/l)** | 0.6 | 1.9 | 0.2 | 0.3 kg ha$^{-1}$ ($PO_4$) |



# The Soil and Water Assessment Tool (SWAT MODEL)

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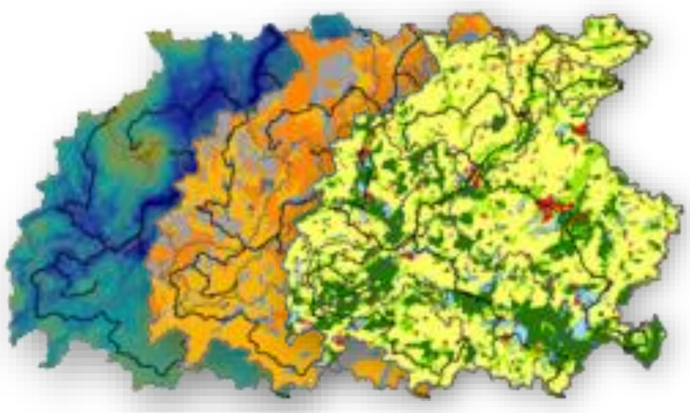
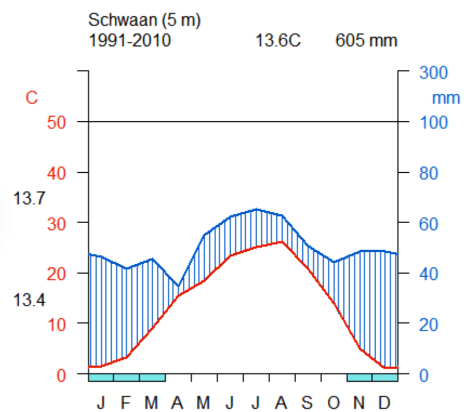
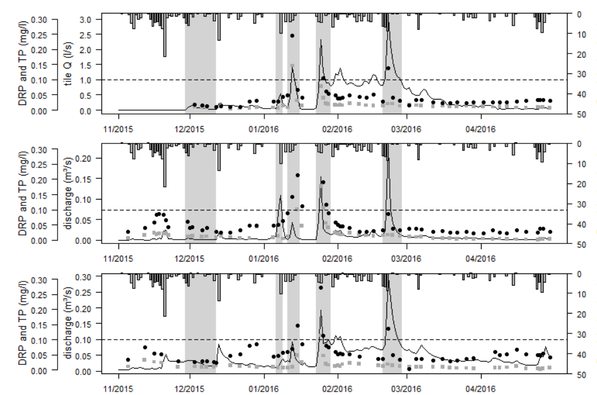
- **Soil and Water Assesment Tool** (SWAT model) to model streamflow and P loads in tile drains
  - Physically-based eco-hydrological model with a **tile-drainage routine**
  - Spatial resolution: HRU (Hydrological Response Unit)
  - Temporal resolution (according to Input data, hourly to yearly)





HRU

Weather data

**Basic model input**

**Discharge (+ nutrient, sediment, contaminant data for model calibration)**

# Evaluating Hydrological Models

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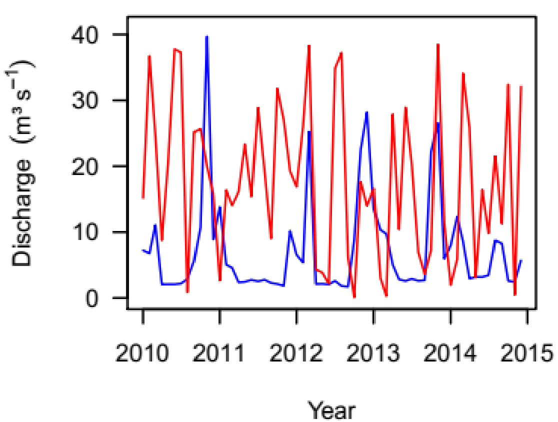
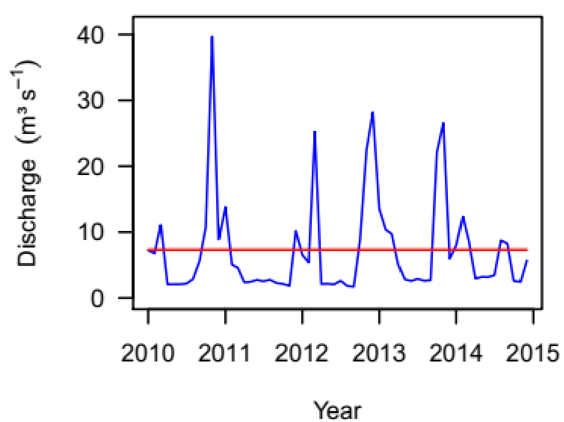
- **Nash Sutcliffe Efficiency** (NSE) as evaluation index
  - Range from – ∞ to 1 (1 is perfect model fit)
  - Values above 0.5 considered as “good”, above 0.75 as “very good”

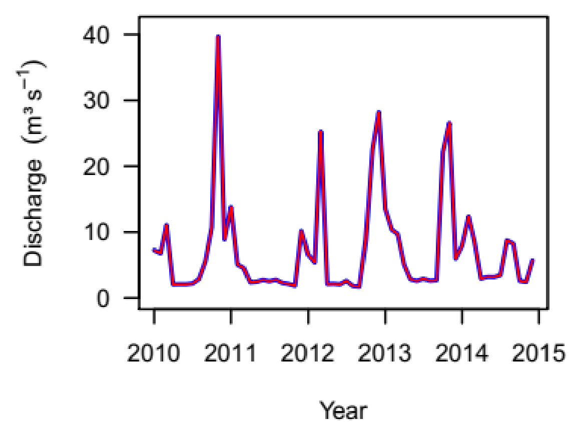
**NSE = -3.96**




**NSE = 0**




**NSE = 1**



# RESULTS

# Calibration of Flow and Phosphorus loads

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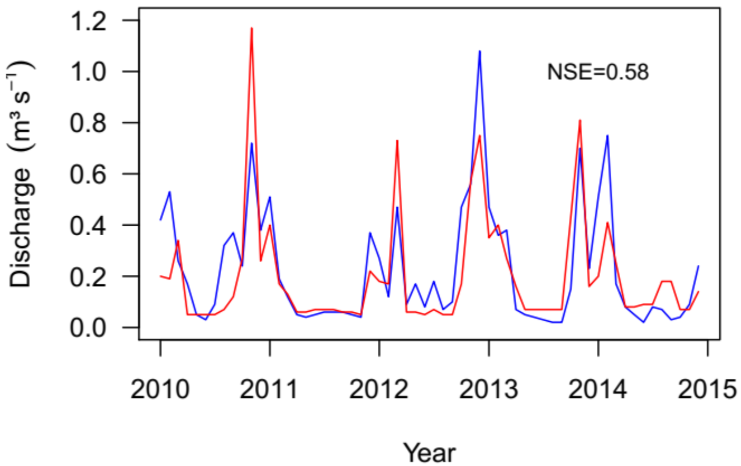
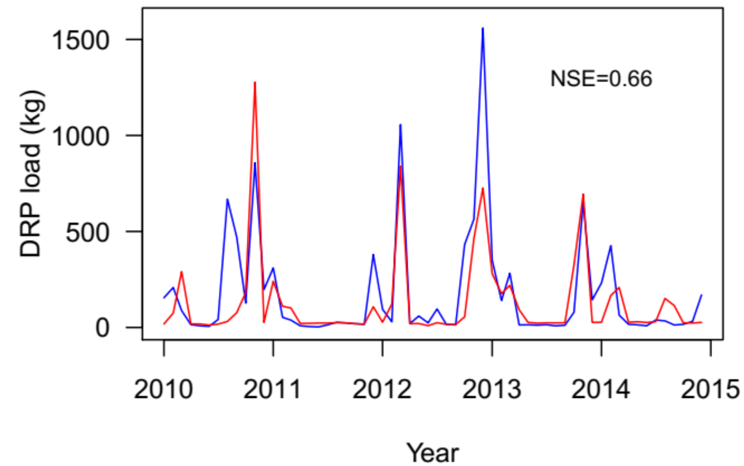
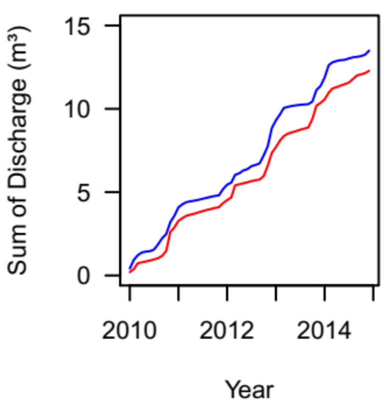
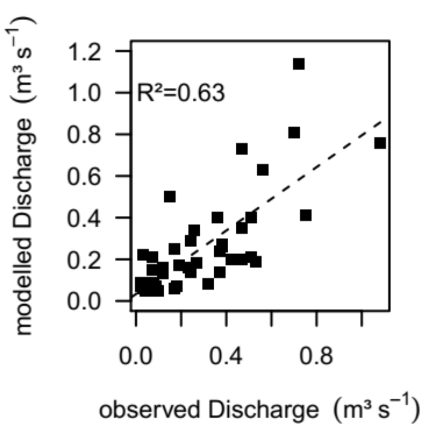
- Measured monthly flow/DRP loads (blue) vs. modelled flow/DRP loads (red) in the calibration period

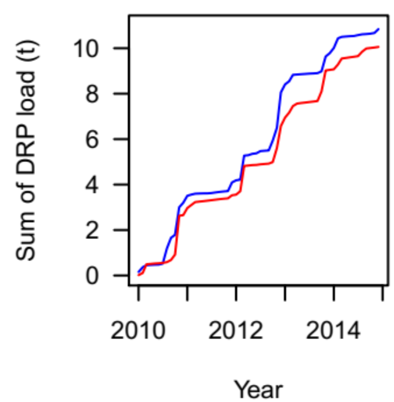


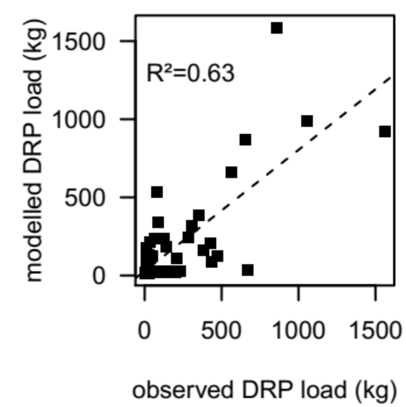




- Sum curves of observed vs. modelled flow and DRP loads and scatterplots of observed and modelled flow and DRP loads in the calibration period



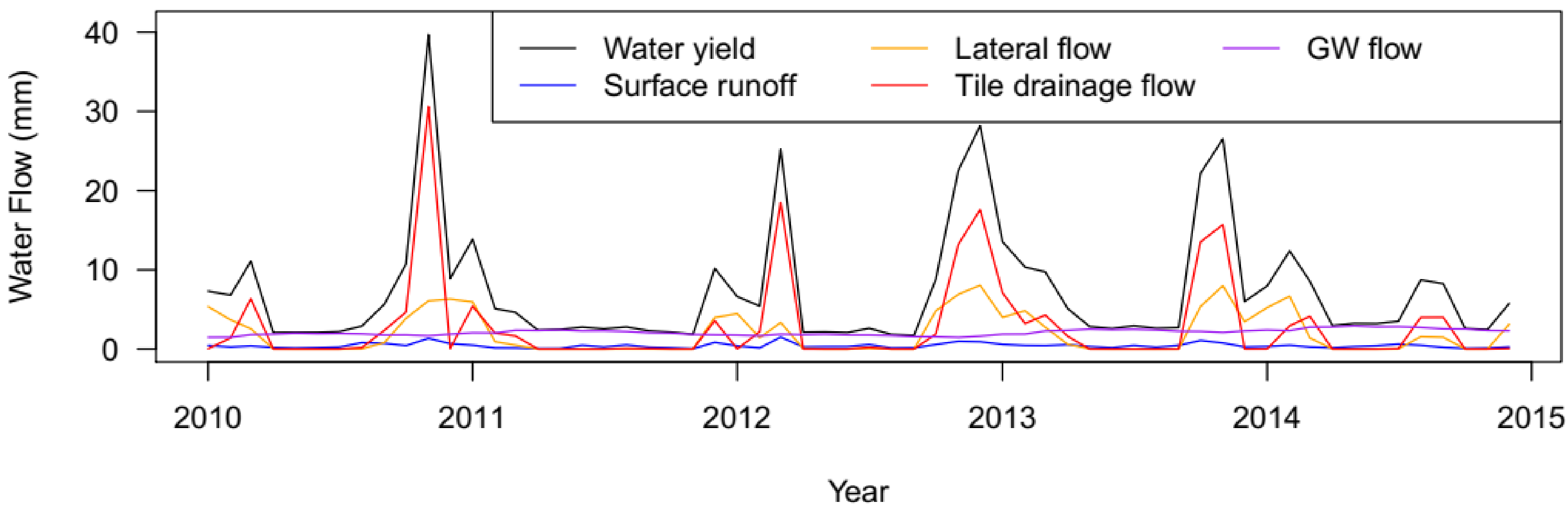



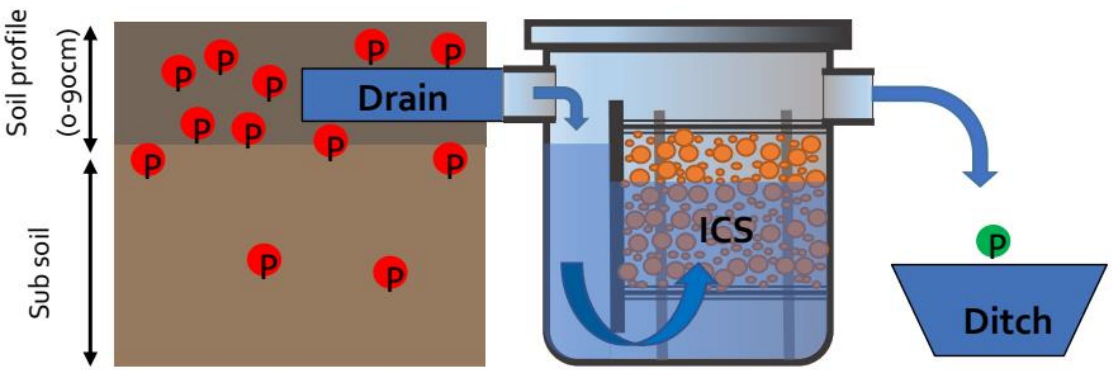



# Calibration of Flow and Phosphorus loads

- Realistic **distribution of flow parameters** is crucial for process understanding and the implementation of scenarios



# Reduction Sceanrios




- **P filters** with iron-coated sand (ICS) in a filter box applied to selected HRUs

- Easy to install
- **High P removal efficiency (80-90%)**
- Low cost installation
- Does not cause any impairment of surface waters

# Reduction Scenarios

- P filters applied to different areas of drained agricultural areas

| | Scenarios | | | | | | |
|---|---|---|---|---|---|---|---|
| **Proportion of area equipped with a filter** | **5** | **10** | **15** | **25** | **50** | **75** | **100** |
| Area (ha) | 310 | 619 | 929 | 1548 | 3095 | 4643 | 6191 |
| Number of drainage plots **(6 ha per collector drain)** | 51 | 103 | 154 | 257 | 515 | 773 | 1031 |
| Annual costs (€) | 16.218 | 32.754 | 48.972 | 81.726 | 163.770 | 245.814 | 327.858 |

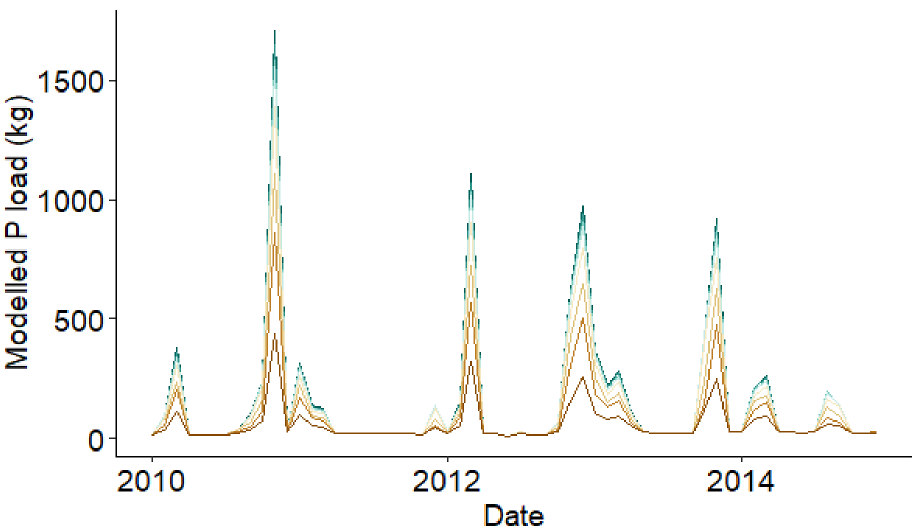
# Reduction Scenarios

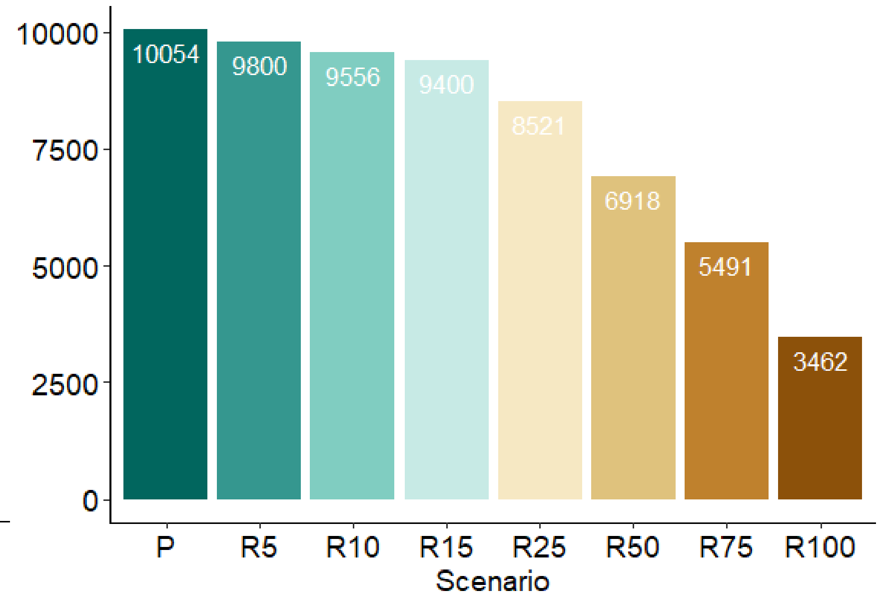
- P filters applied to different fractions of drained agricultural areas

| | **Base Model** | | **Reduction Scenarios** *(percentage of agricultural area equipped with P filter)* | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | **obs P** | **mod P** | **5** | **10** | **15** | **25** | **50** | **75** | **100** |
| **P load (kg)** | 10841 | 10054 | 9800 | 9556 | 9400 | 8521 | 6918 | 5491 | 3462 |
| **P load reduction (kg)** | | | 254 | 498 | 654 | 1533 | 3136 | 4563 | 6592 |
| **Reduction (%)** | | | **3** | **5** | **7** | **15** | **31** | **45** | **66** |

# Reduction Scenarios

- The installation of P filter may cause a 66% reduction of the total DRP load



# Outlook

- Long-term studies on in-situ filter techniques will improve the development and implementation of scenarios to hydrological models
- In-situ tests of different filter materials will help getting a wide range of scenarios of P reduction

- Using the same approach for developing N reduction scenarios

# Thank you for your attention

# Q & A

# Next seminars

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- Friday 2/10 – 10h – 11h30:

**Filter technologies for N removal from agricultural waters**

https://northsearegion.eu/nuredrain/

# Acknowledgements

Vlakwa
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WATER. VANDAAG EN MORGEN.

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Rostock
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Oost-Vlaanderen
Voor ieder van ons

west-vlaanderen
de gedreven provincie