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Recovery of phosphorus by chemical treatment

Nico Lambert – KU Leuven
Process & Environmental Technology Lab

KU LEUVEN

Introduction

Relevant research question:

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

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
- A desorption process using an **alkaline** solution is proposed without harming the adsorbing material.

Integration of P-adsorbing material in a circular process

Iron Coated Sand (ICS)
DiaPure®
Vito A & B
FerroSorb SW

Introduction

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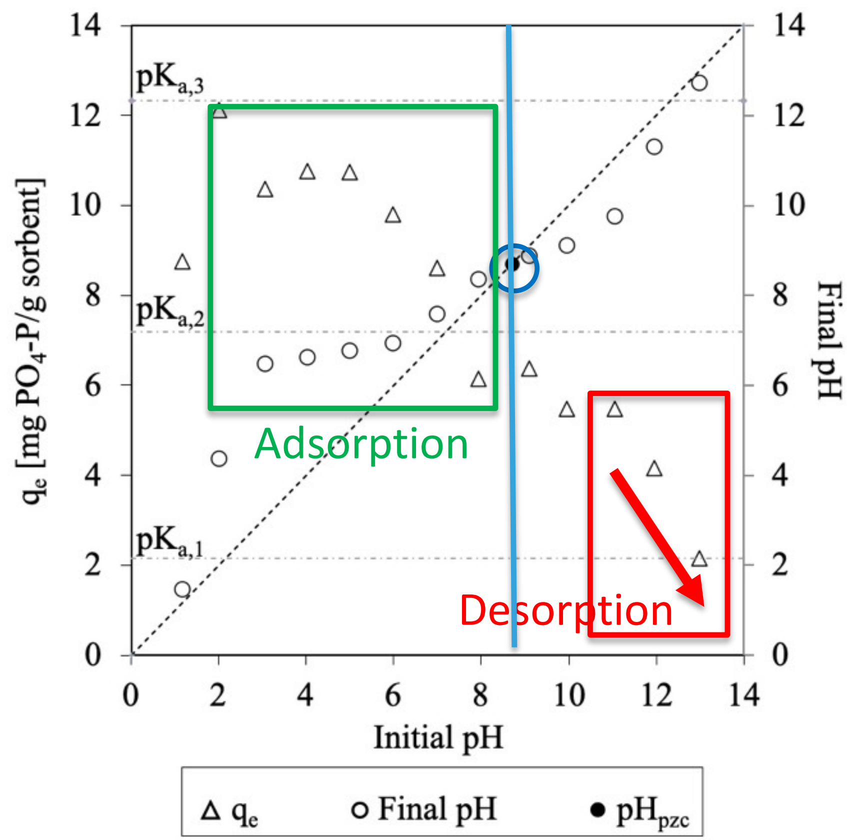
Theoretical basis:

- The influence of initial pH on the adsorption capacity q_e for Fe and Al based adsorption materials
- Adsorption/desorption are **balancing processes** until an equilibrium is reached!

- pH 8.7 = pH_{PZC}
  = final pH is equal to the initial pH
- pH range 2 - 8.7: high q_e
- pH range 8.7 – 13: low q_e
- pH>11 the q_e drops considerably



Concept of alkaline desorption

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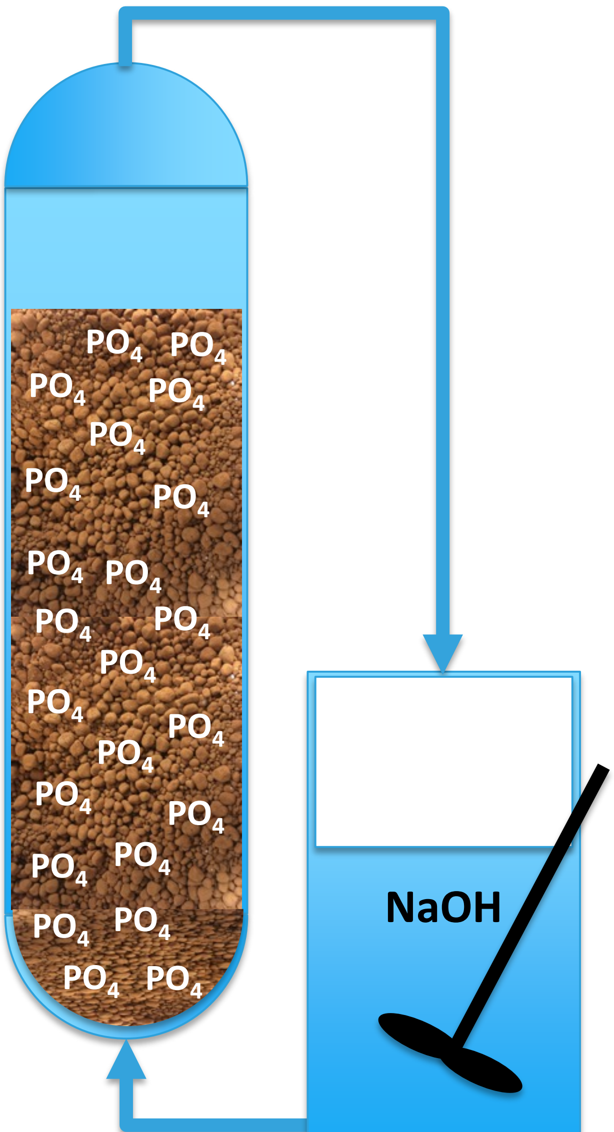
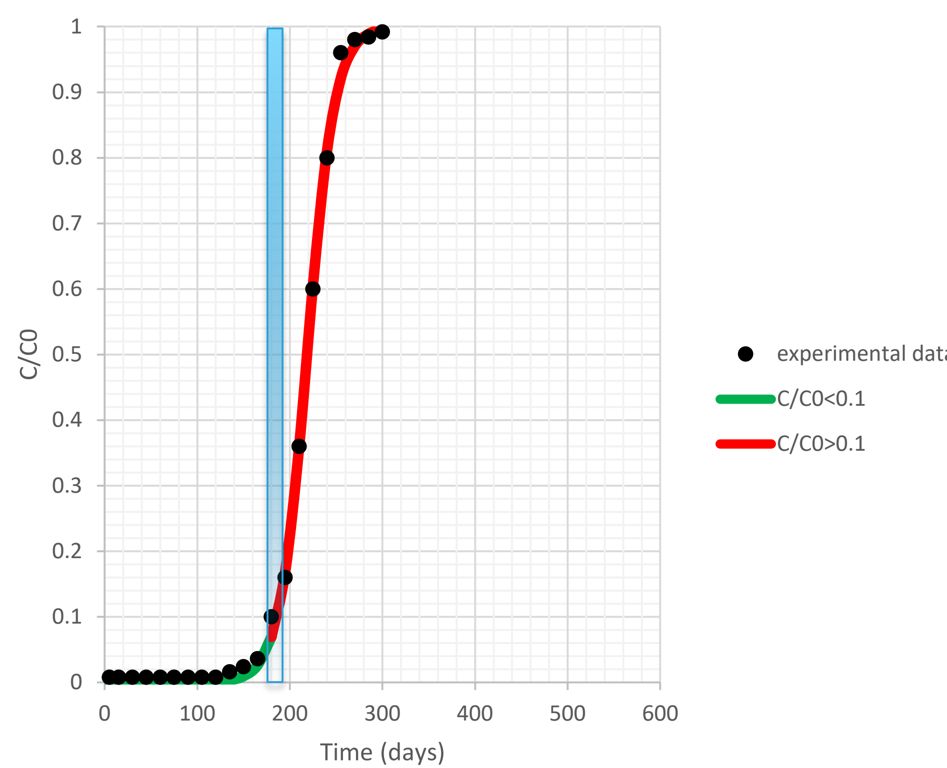
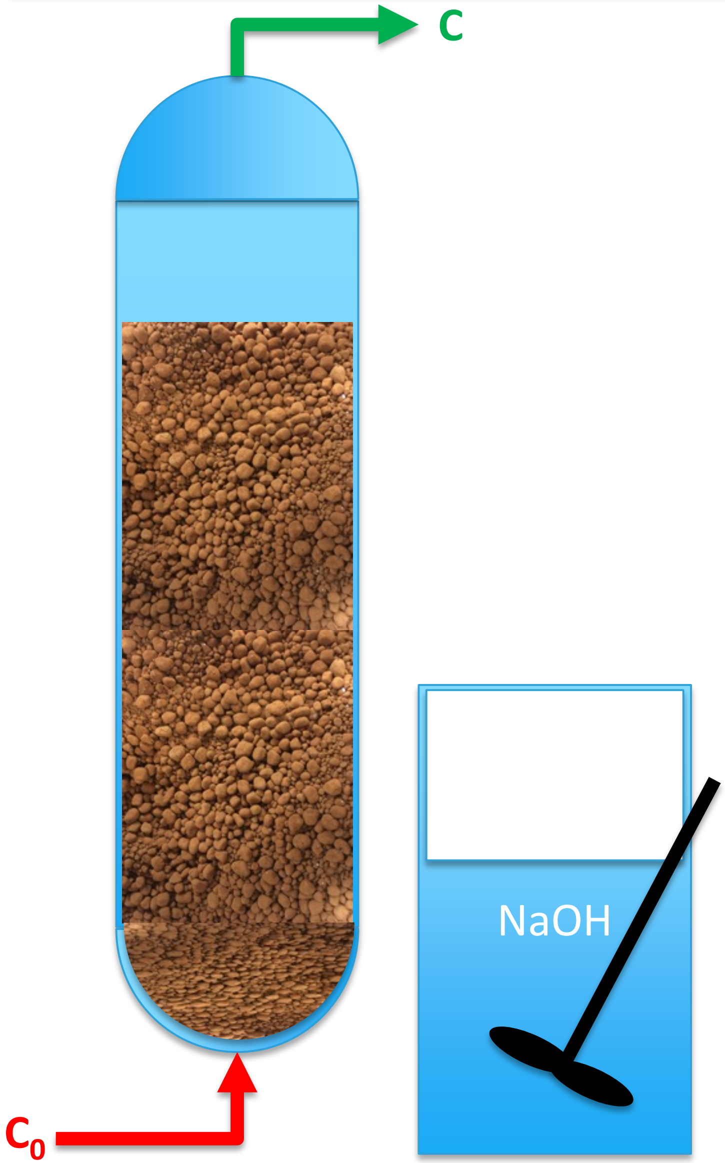


Desorption Phase @ C/C0= 0.1

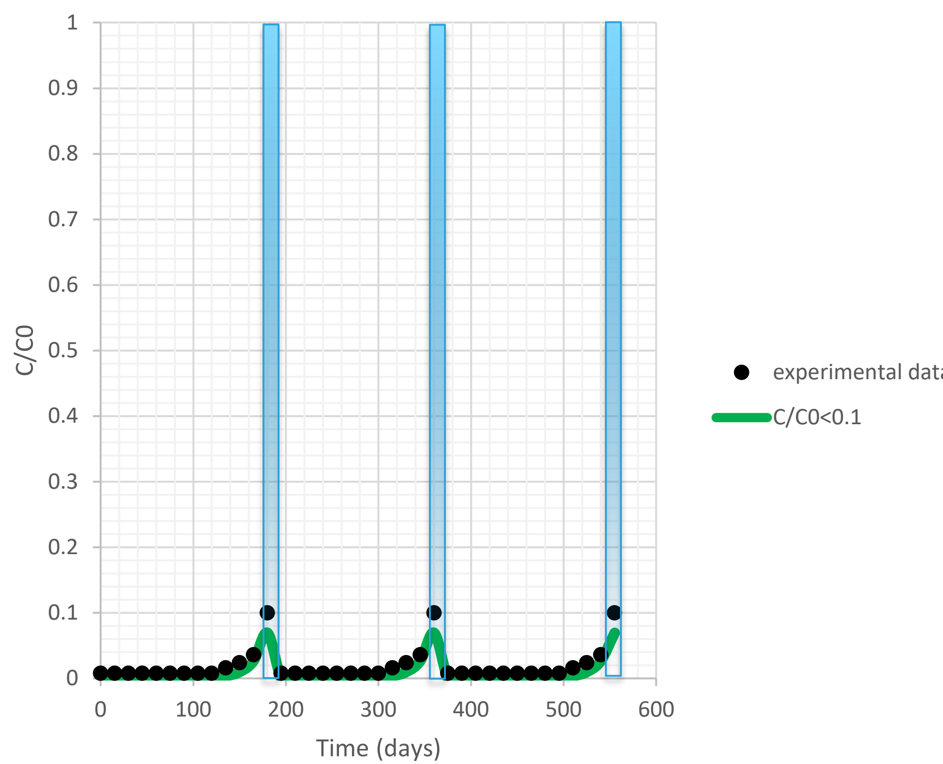


Concept of alkaline desorption



Regeneration of the saturated sorbent and recovery of phosphorus

Intermittent regeneration of ICS



Materials & Methods

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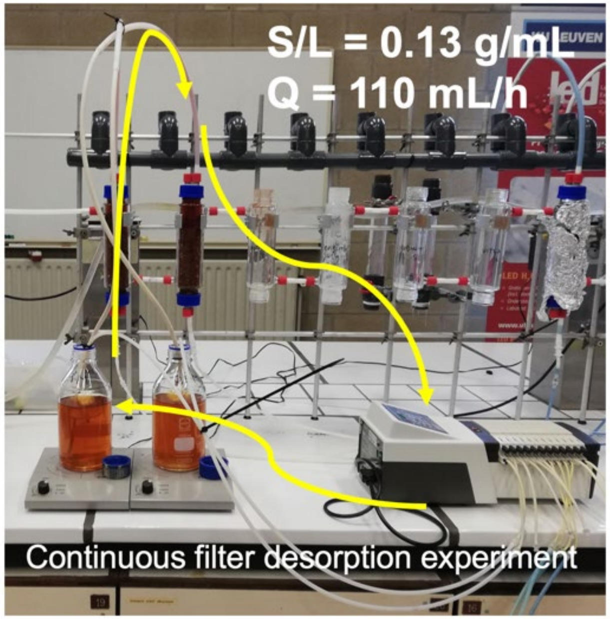
1. **Batch desorption experiments:** 5g of pre-dried saturated **ICS** was brought into contact with NaOH solution.

   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 ad- & desorption experiments:** 1 liter of NaOH solution was recirculated over an adsorption column filled with 150 cm^3 of saturated adsorption material.



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

Results & Discussion
Batch experiments

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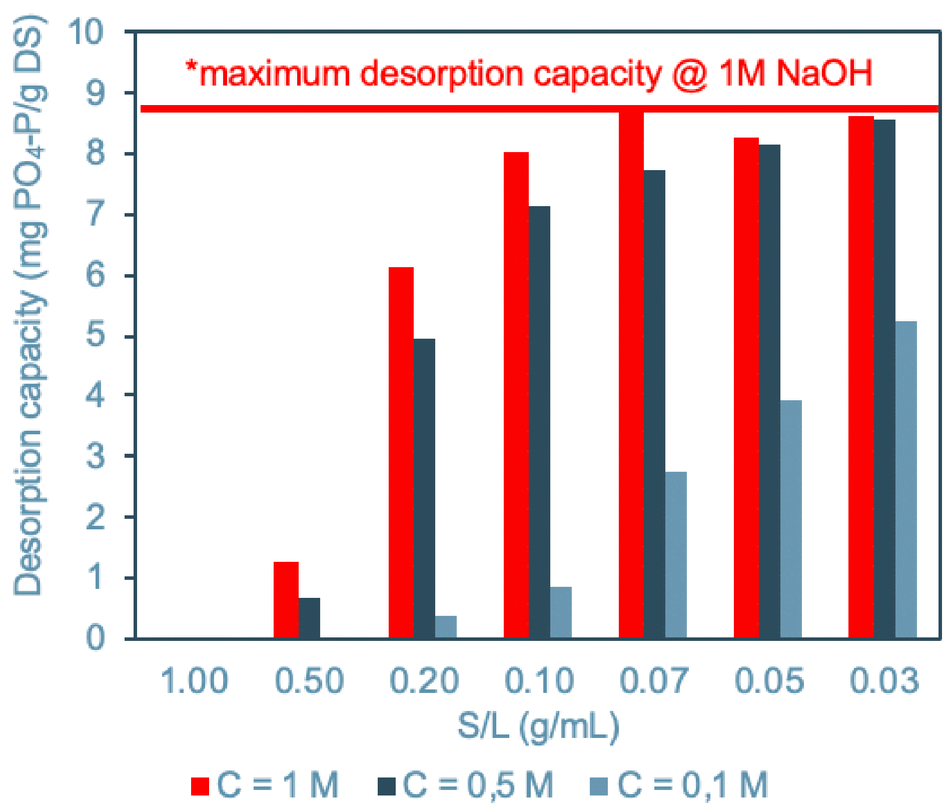
- The composition of 1 g of saturated ICS granules was determined by a complete destruction of the granules by Aqua Regia and ICP analysis:
  - Phosphorus: 15.30 +/-1.25 mg P/g DS **=1.5%P**
  - Iron: 590.7 +/-8.7 mg Fe/g DS **=59%Fe**

- Optimal NaOH concentration = 0.5 M
- Optimal contact time = 24 h or more
- Optimal S/L ratio = 0.10 - 0.05 g/mL
- P-desorption efficiency > 50% @ 0.5 and 1 M NaOH



Results & Discussion
Continious filter experiments: Adsorption

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- The breakthrough curve of ICS 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.

Regeneration is highly appropriate in the case of a short EBCT

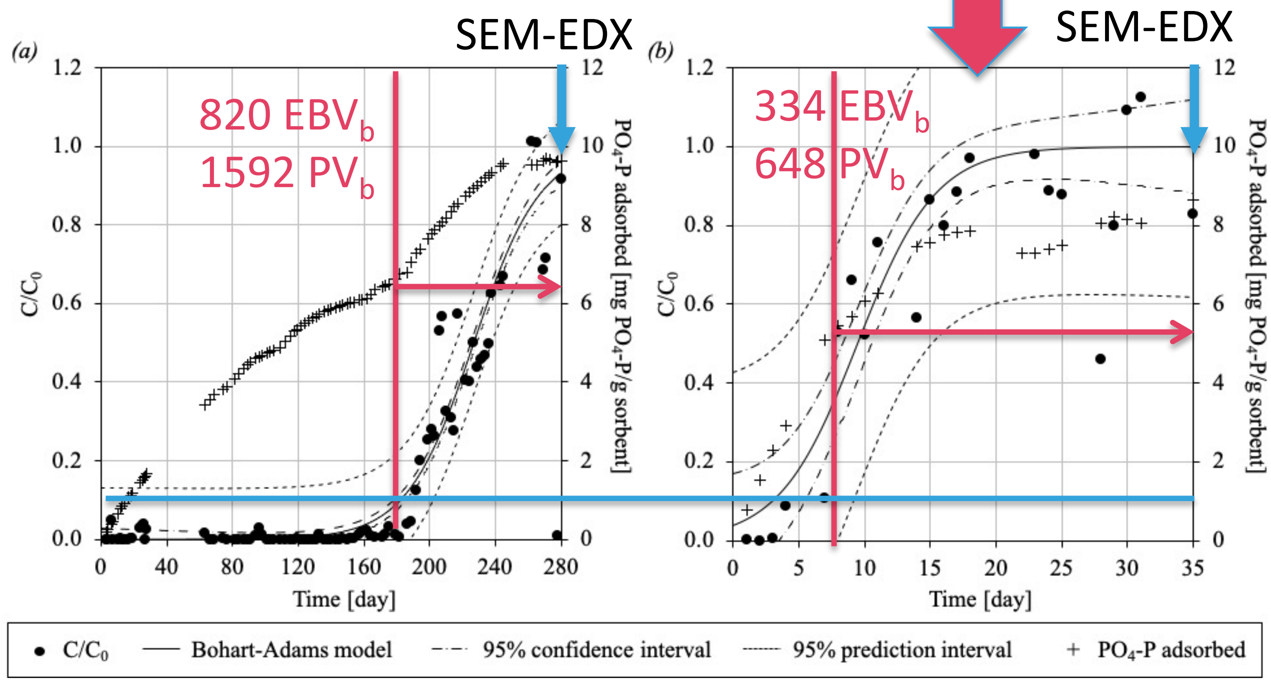
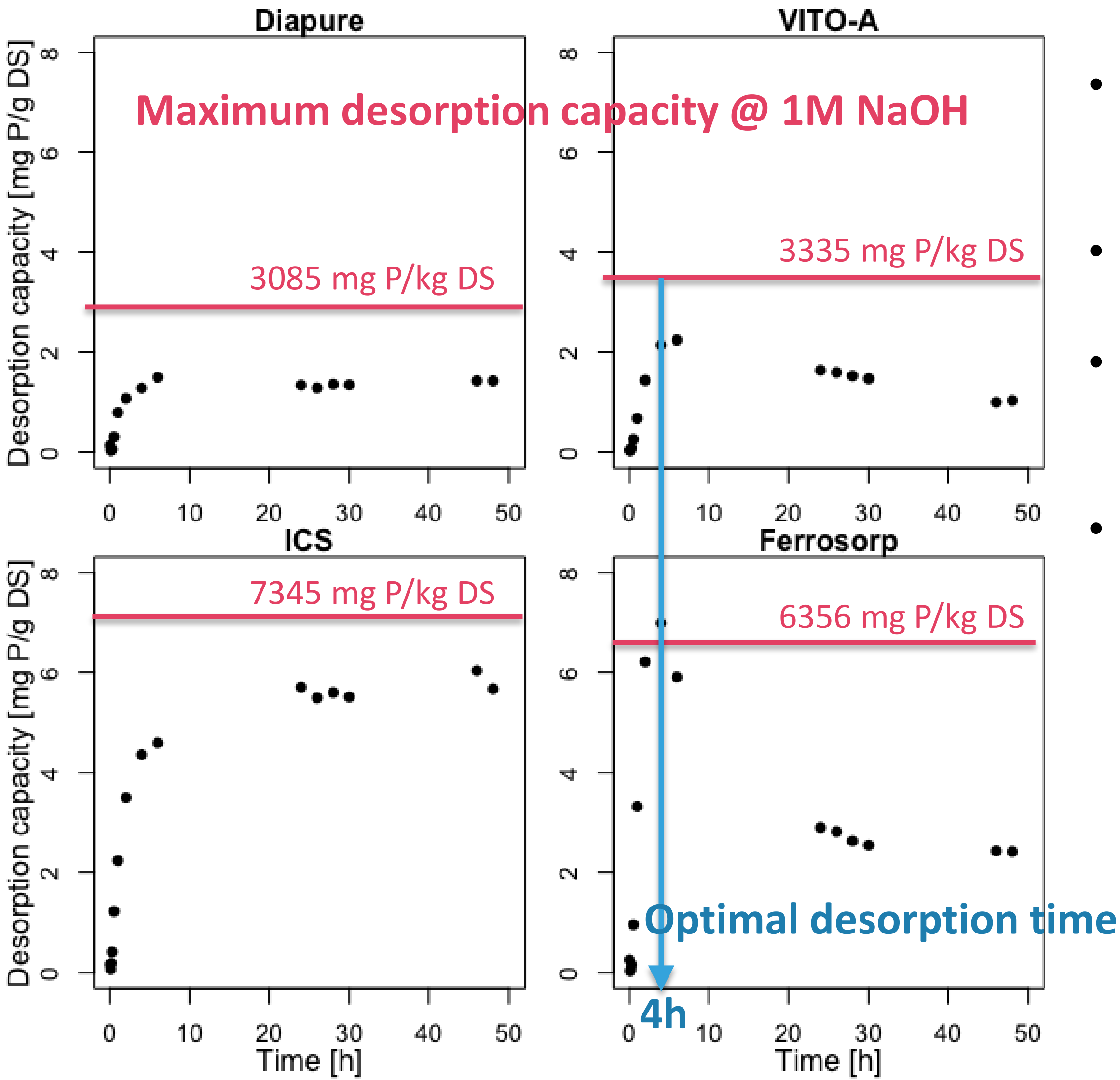


Figure: ICS adsorption column experiments on lab-scale (influent P concentration = 25 mg PO_4-P/L) with EBCT= 5.5 h (a) and EBCT= 0.5 h (b)

Results & Discussion
Continious filter experiments: Desorption



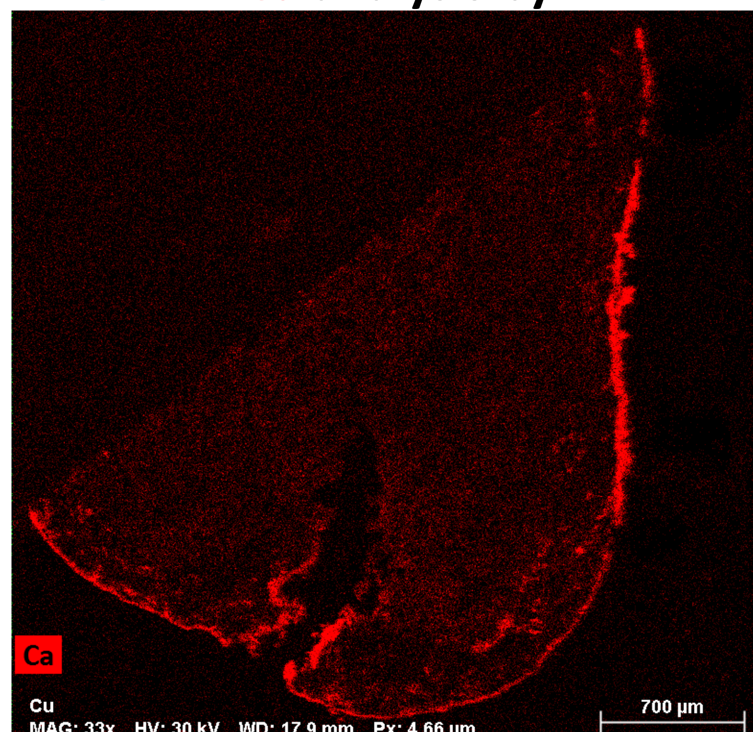
- Continuous desorption experiment in recycle
- NaOH concentration = 0.5 M
- Optimal desorption time = material dependent
- P-desorption efficiency > 50% @ 0.5 NaOH

Results & Discussion
SEM-EDX analysis @ EBCT of 0.5 h

- SEM-EDX of saturated DiaPure® of column experiment with **EBCT of 0.5 h**.
  - The phosphate is mainly adsorbed at the outer layers of granules.
  - Calcium forms deposits on the adsorbent surface and disturb the alkaline desorption.
  - Acid regeneration step before alkaline desorption?

polished DiaPure® granule embedded in a resin

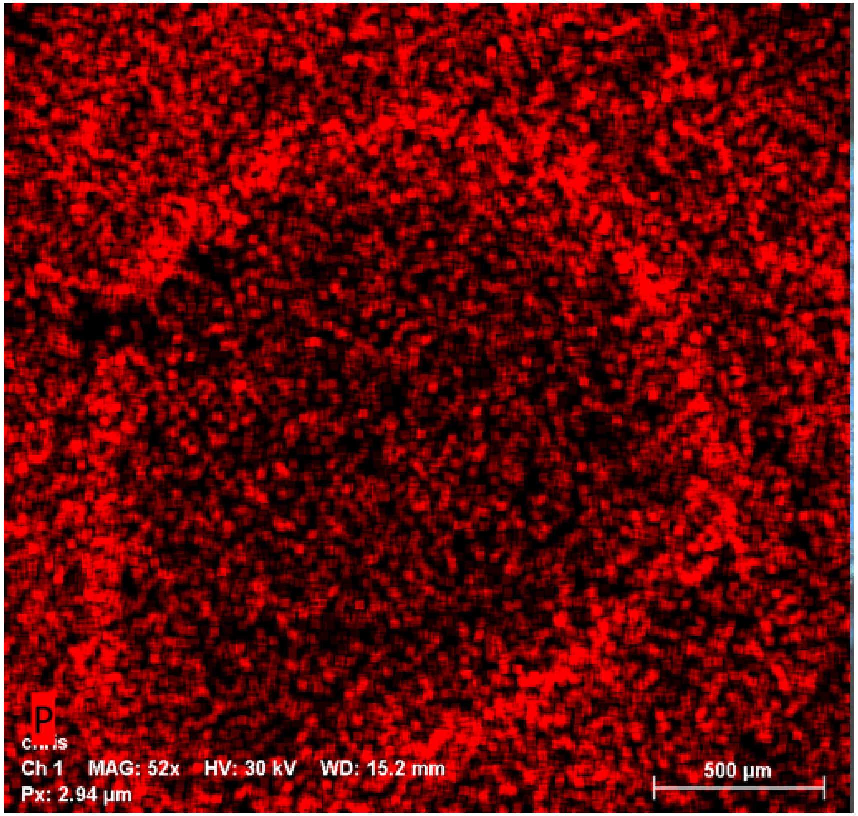
Fe – P – Ca analysis by EDX



Results & Discussion
SEM-EDX analysis @ EBCT of 5.5 h

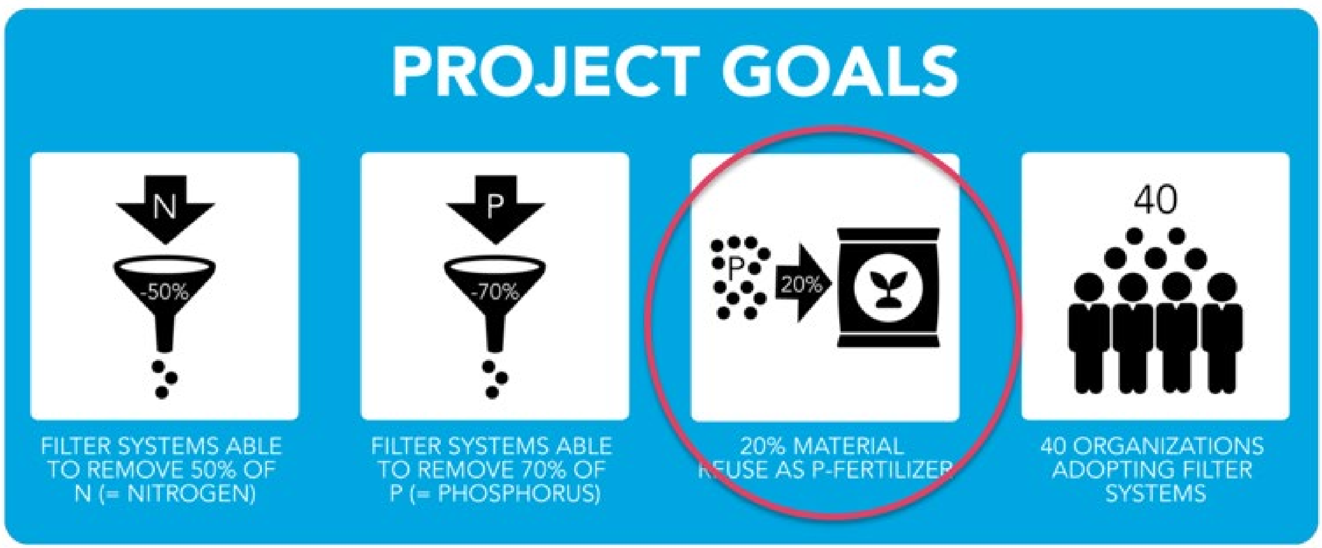
- SEM-EDX of saturated ICS of column experiment with **EBCT of 5.5 h**.
  - Phosphorous is accumulated at the sand core of the granule.
  - Phosphorous migrates towards the core of the granule.

Si – Fe – P analysis by EDX



Conclusions

- Optimal NaOH concentration = 0.5 M
- Optimal desorption contact time = material dependent
- P-desorption efficiency > 50% @ 0.5 M NaOH
- Leaching of Fe during the desorption process is a problem
- Desorption of P from the inner layers of the granule will be difficult
- Calcium deposits should be avoided by an acid wash



Q & A

Part IV: Nutrient removal modelling

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Nutrient reduction potential using end-of-pipe solutions for an entire catchment

Andreas Bauwe, Bernd Lennartz – University of Rostock

#EUGreenWeek

2021 Partner Event

+++ Filter systems for nutrient removal from agricultural waters +++

1 June 2021

The Warnow river basin

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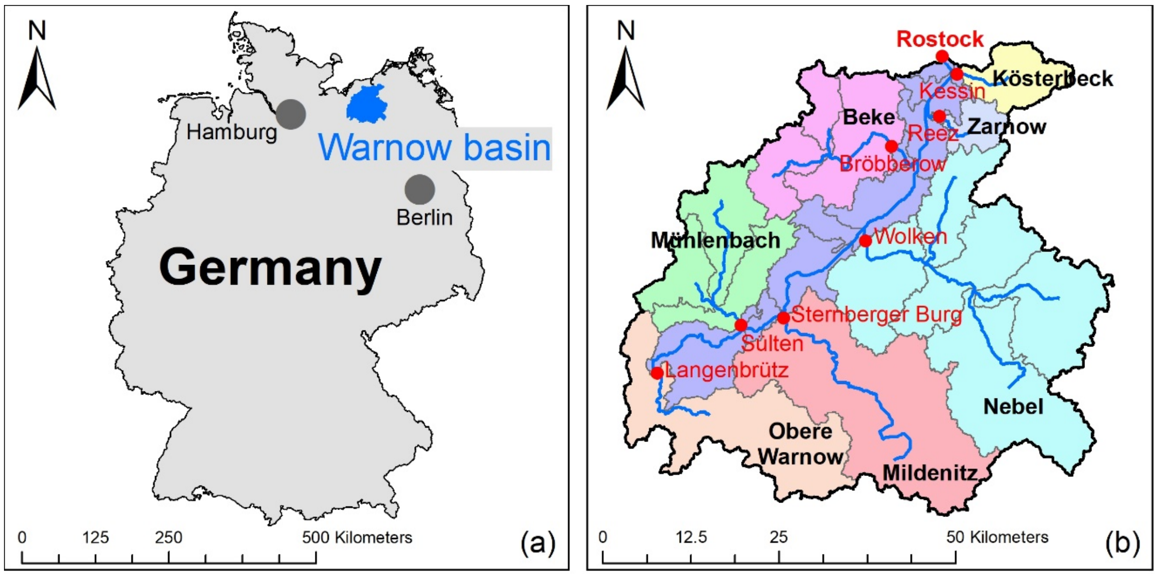
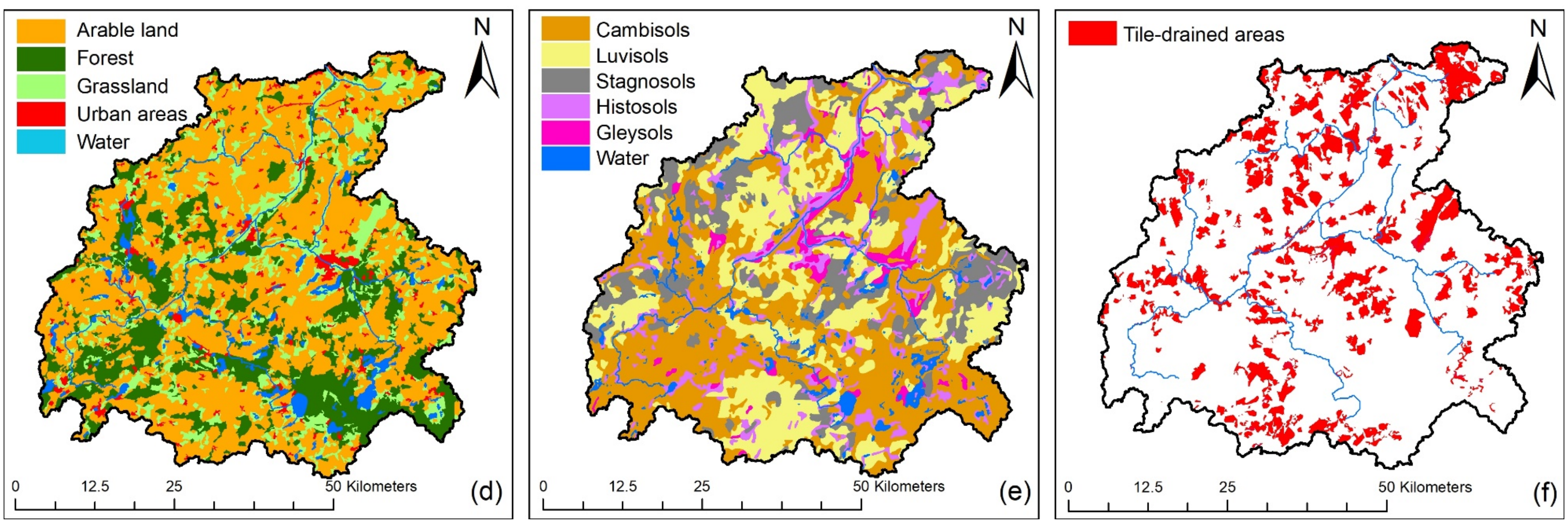


Size: ca. 3,000 km² (second largest German watershed that discharges into the Baltic Sea)

Land use: Arable land (57%), Forest (21%), Pasture (15%)

Soils: Cambisols, Luvisols

Tile-drained areas: 19%



Background

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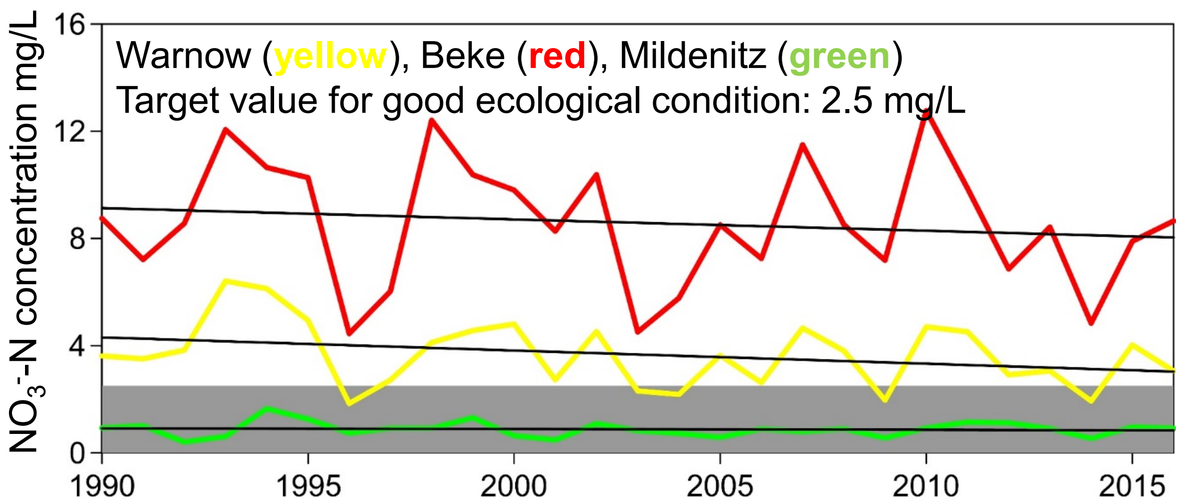

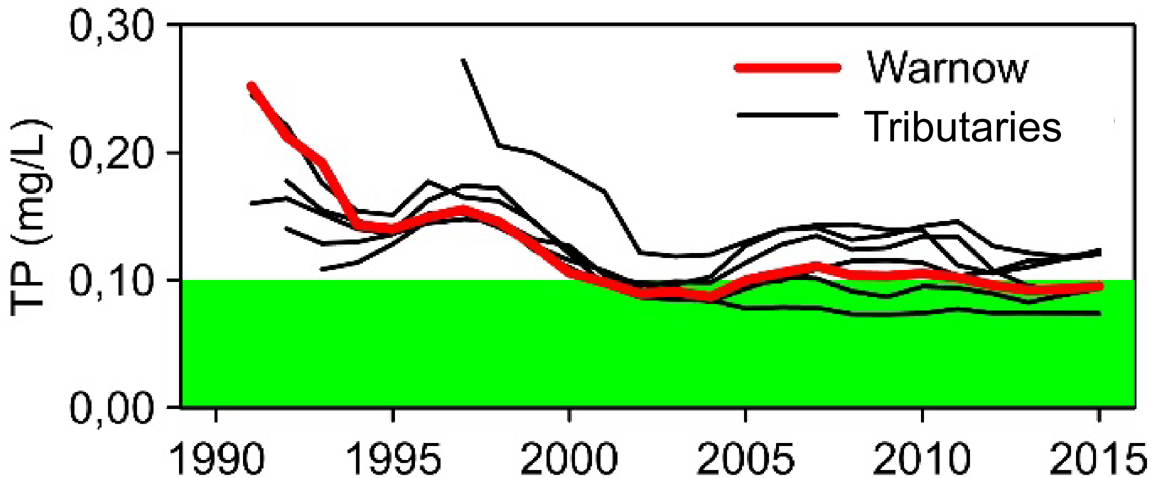

- Slow decrease of NO_3^--N concentrations during the last 30 years
- Large differences in NO_3^--N concentrations among the sub-basins depending on land use
- Mitigation measures needed for sub-basins dominated by agriculture
- Strong decrease of TP concentrations in the early 1990s mainly due to improved treatment of wastewater
- Target values for TP are complied in most sub-watersheds
- However: HELCOM demands a reduction 110 t TP/a for Germany

Reduction measures needed for N + P (end-of-pipe)

Methods

End-of-pipe solutions to reduce nutrient loads in tile-drained areas

Phosphorus
Filters

Nitrogen
Constructed wetlands

Modeling the reduction potential using the SWAT model

Model input

- Digital Elevation model,
- Weather data
- Land use
- Soil data
- Land management
- Vegetation
- ….

Modelling approach

1. Calibration and validation of stream flow
2. Calibration and validation of P and N loadings
3. Implementation of filters and constructed wetlands in the model

Reference simulation

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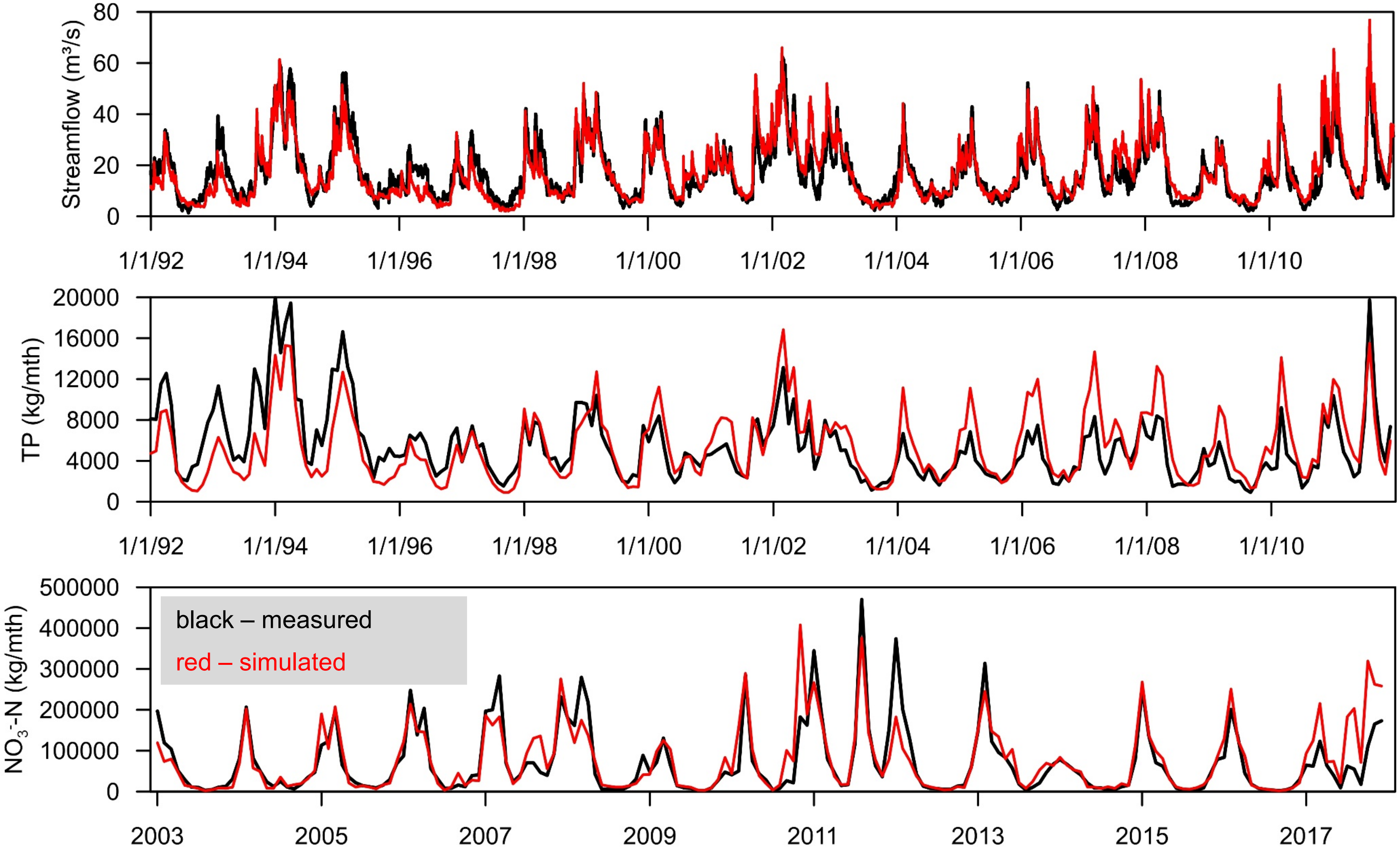

P reduction scenarios

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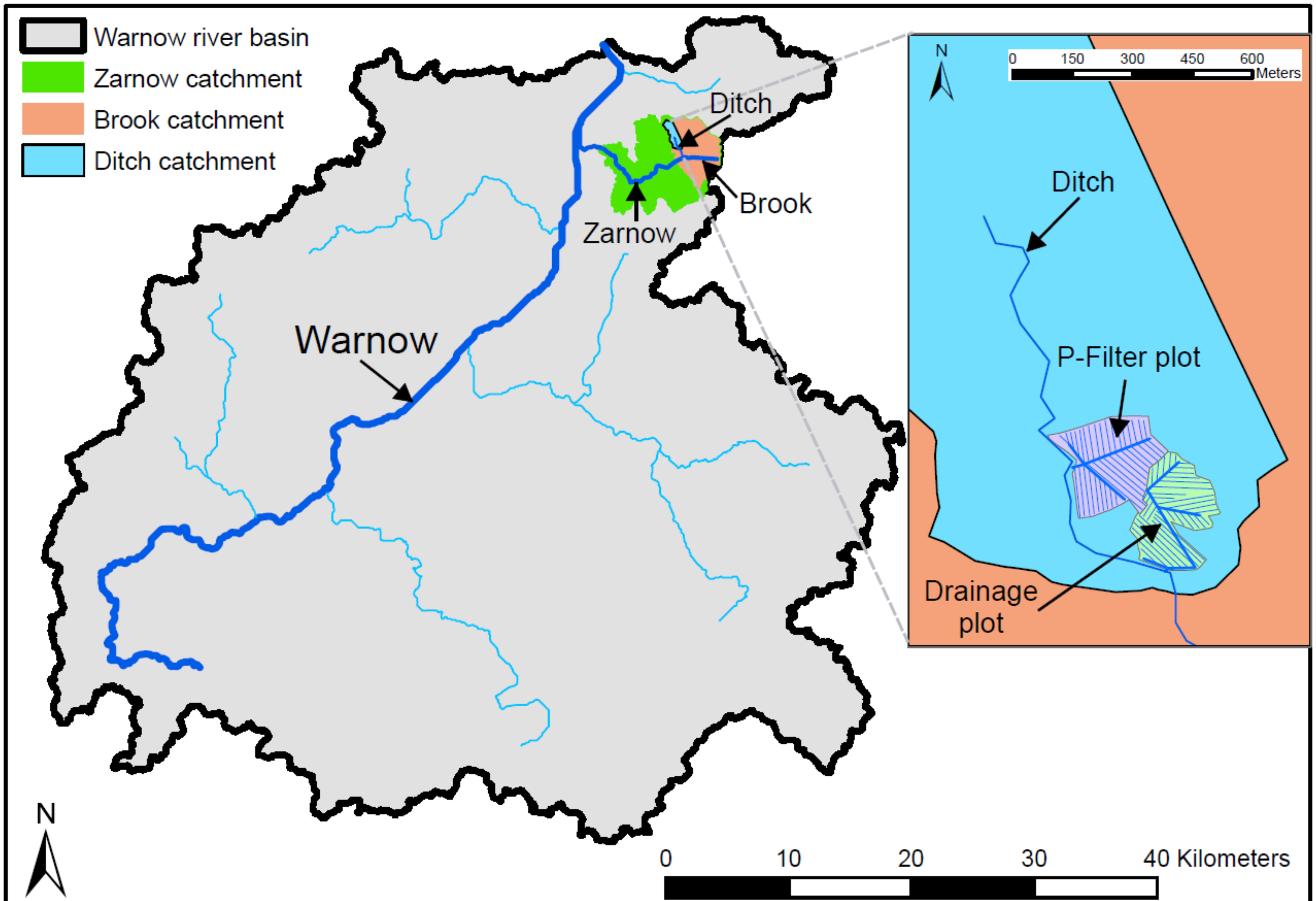
Evaluation of P filters in tile-drained areas at different spatial scales



P reduction scenarios

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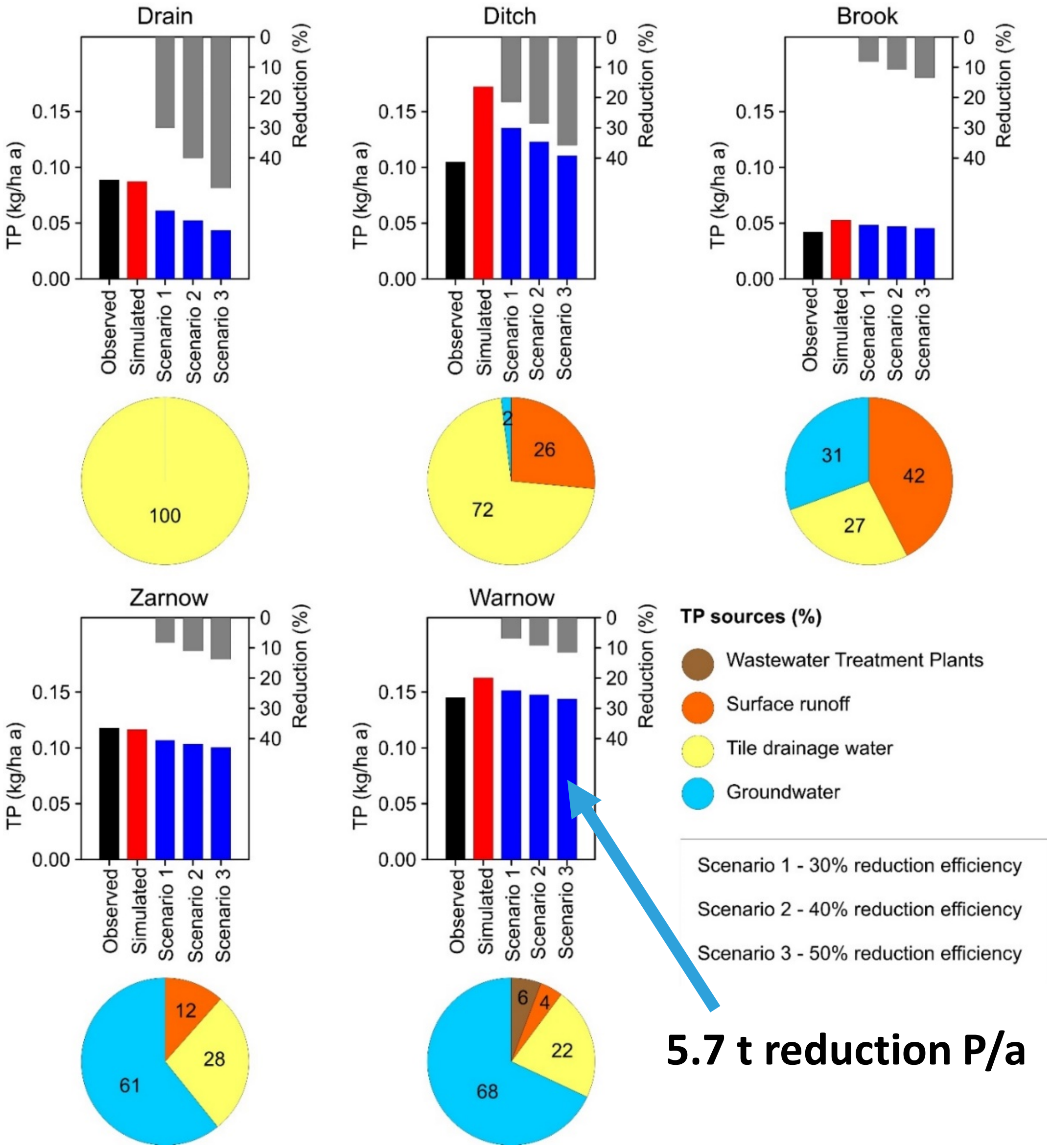

- Good fit of measured and modeled values at different spatial scales.
- Effect of P filters at catchment scale depends on proportion of tile-drained areas.
- P filters could contribute to reduce P losses notably in the Warnow river basin.

N reduction scenarios

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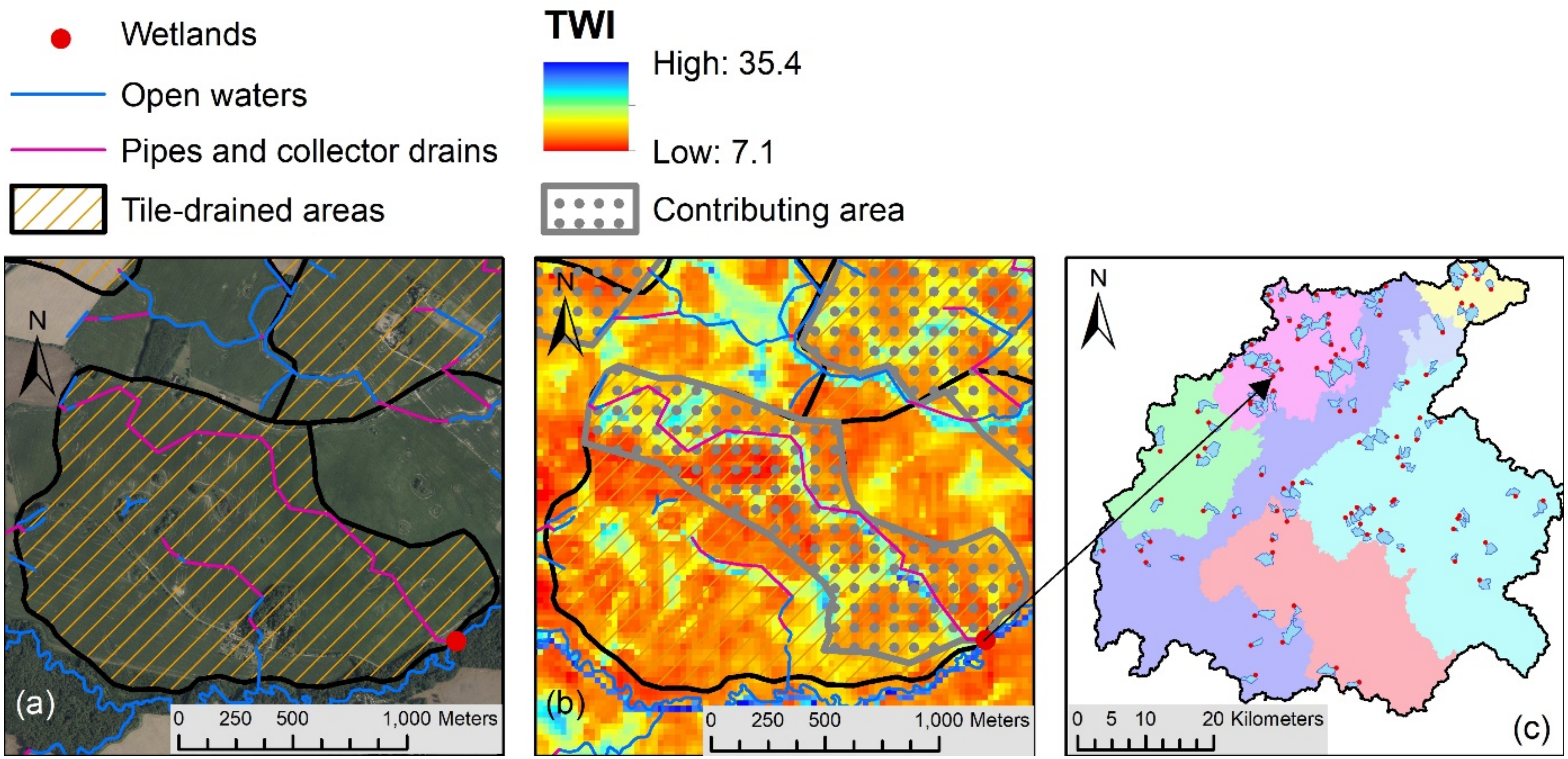
Evaluation of constructed wetlands in tile-drained areas



- Contributing areas were identified by using maps of tile-drained areas, running waters (open or as pipes) and aerial photographs.
- Constructed wetlands (CWs) were placed in moist areas according to topographic wetness index (TWI).
- 97 suitable spots for CWs were identified.

N reduction scenarios

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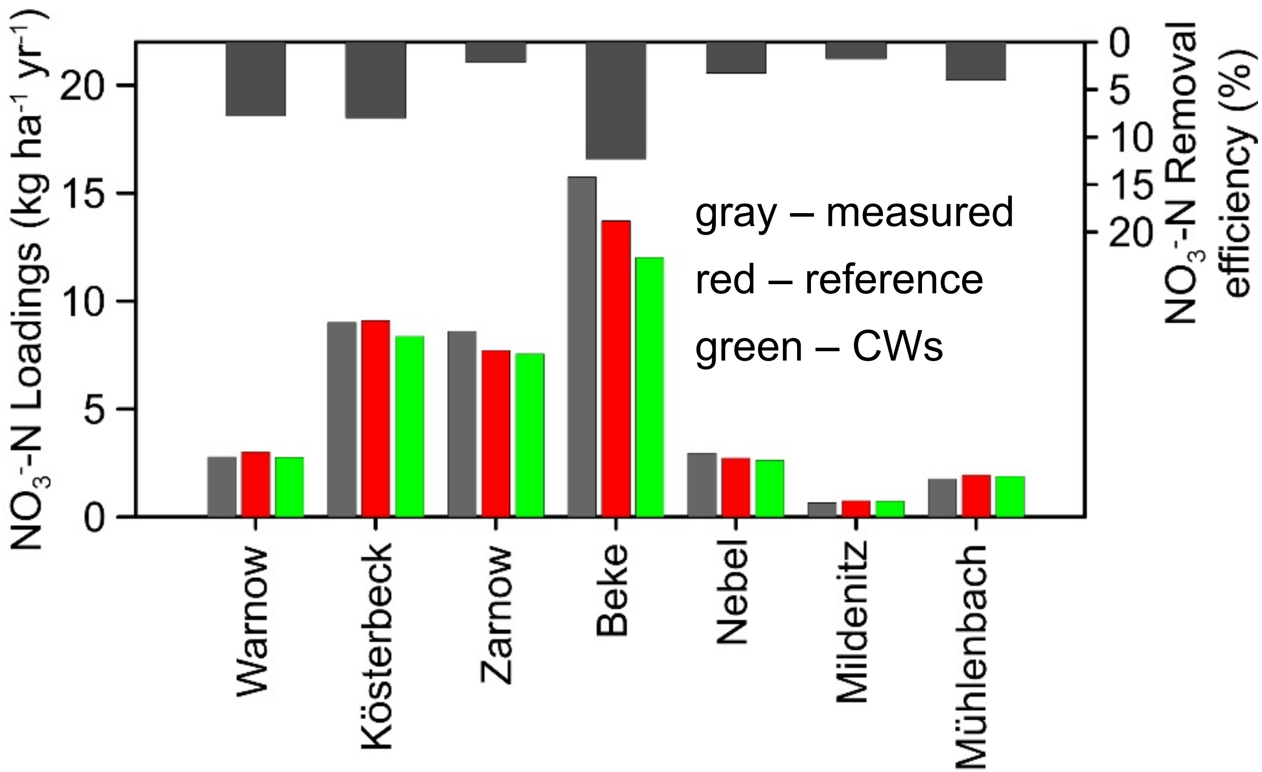

- Measured NO_3^--N loadings were reproduced well by the model.
- The implementation of constructed wetlands had positive effects on the surface water quality with an overall NO_3^--N removal efficiency of 7.8%.
- The NO_3^--N removal efficiency depended on subbasin characteristics (number of CWs, ratio between contributing area and subbasin area).

N reduction scenarios

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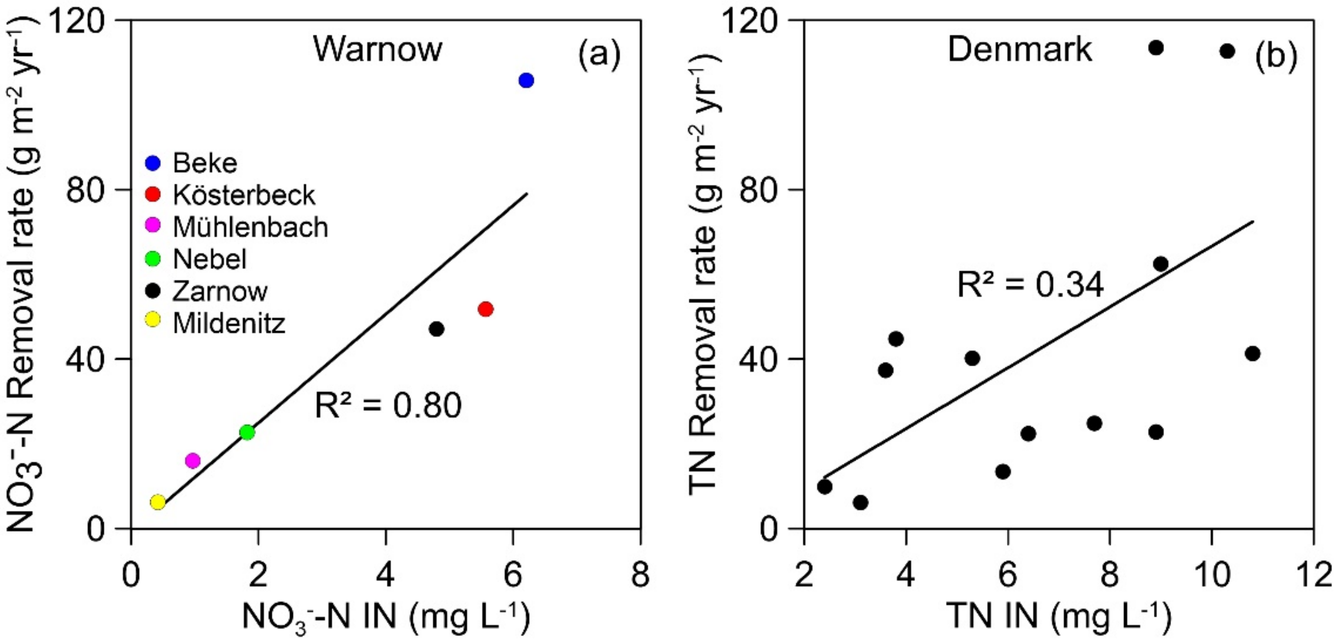

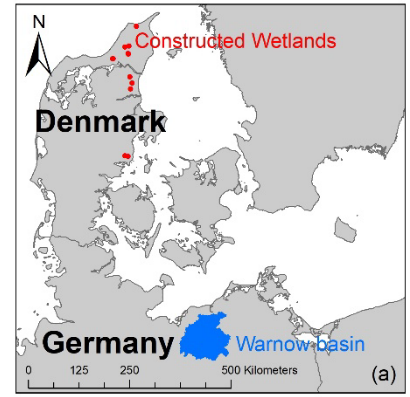

- The scenario results were verified by comparing simulation data with recordings of 13 existing CWs in Denmark (thanks to the Danish partners for providing the data!).
- The NO_3^--N removal rates for the Warnow basin and CWs in Denmark were similar.
- Both for the Warnow basin and the CWs in Denmark, there was a significant positive relationship between input concentration and removal rate.
- Due to site-specific characteristics, this relationship was weaker for the Danish CWs.

Summary

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- Through the widespread installation of filters in tile-drained areas, the TP loads in surface waters could be reduced by 5.7 t yr^{-1}, which corresponds to an overall reduction of ca. **10%**.
- The effect of P filters on a catchment scale depends on proportion of tile-drained areas.
- NO_3^--N loads could be reduced from 900 t yr^{-1} to 840 t yr^{-1}, which corresponds to an overall reduction of ca. **8%**.
- NO_3^--N removal rates varied strongly among the subbasins ranging from 6 to 106 g m^{-2} yr^{-1} and they were positively correlated with the input concentrations.
- The installation of filters for P reduction and constructed wetlands for N reduction should be prioritized, focusing on hot-spot areas, in which the largest benefit is expected.

Thank you!

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Cost-effectiveness of the filters and the farmers' opinion

Charlotte Boeckaert, Vlakwa

P removal

Drainage water

P filterbox

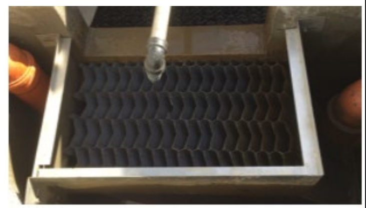
80 – 100%

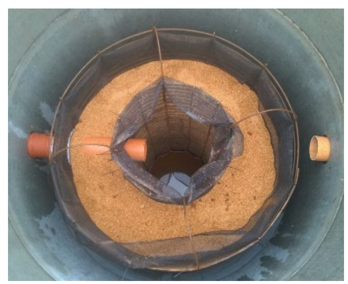
Inline P filter

Organic matter

50 – 80%

Sediment + reactive P filter





Sediments

ongoing

0,1 – 0,5 mg P/l

Greenhouse effluent

DIY

99%

Company

99%

10 – 20 mg P/l

Cost P filter

Water	Filter	CAPEX	OPEX	Yearly cost	Total P removal (kg P)	Cost effectiveness (€/kg P)
Drainage (0,25 mg P/l)	P filterbox	€ 635	€ 19	€ 78,2	0,06	**1 264**
	Drainage water (0,46 mg P/l)				0,19	409
	Drainagewater (0,12 mg P/l)				0,02	4 938
Greenhouse (15 mg P/l)	DIY	€ 690	€ 95	€ 164	1,94	**85**

Cost effectiveness P-filter

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FL – Measures Cost Model

Measure	€/kg P
DIY	85
Non-turning soil tillage	174
Green cover	284
Municipal WWTP	363 - 1006
P filterbox	1264
Buffer strips	2160
Individual WWTP	5235 - 5913

N removal

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Drainage water

MBBR Subsoil

60%

MBBR Containerized

75%

ZVI

90%

10 – 40 mg N/l

Greenhouse effluent

DIY

85%

50 – 100 mg N/l

Cost N filter

	Application	CAPEX	OPEX	Yearly cost	Total N removal (kg P)	Cost effectiveness (€/kg N)
DIY	Greenhouse effluent	€ 2 700	€ 1 400	€ 1 600	12.44	128.76
Subsoil	Drainage	€ 30 000	€ 2 900	€ 5 550	52.84	105.06
Containerized	Drainage Off-grid	€ 50 000	€ 2 700	€ 7 180	71.11	101.01
	Drainage	€ 40 900	€ 3 800	€ 7 460	71.11	104.97

Cost effectiveness N-filter

FL – Measures Cost Model

Measure	€/kg P
Green cover	3
Municipal WWTP	59(-163)
Reduced fertilization	70
MBBR	101-129
Individual WWTP	378-427

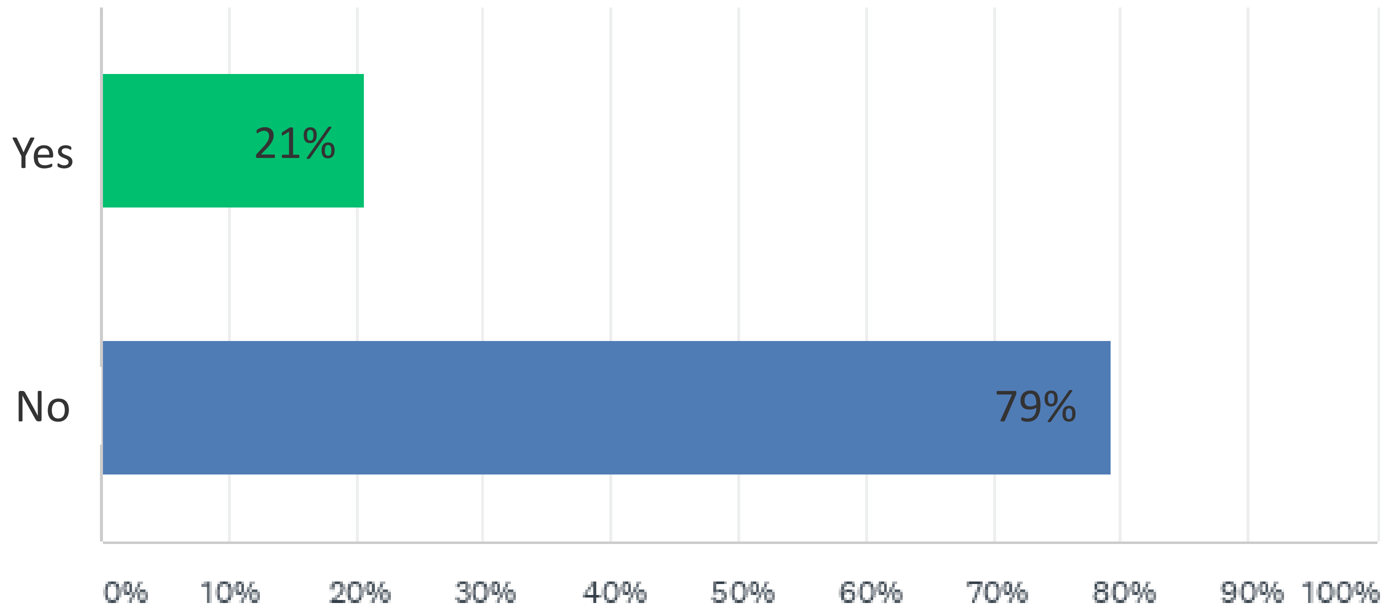
Farmey Survey – FL - Greenhouses

- Which requirements should the filter have?
- Are individual or collective filters recommended?
- Who should pay for these filters?

→ 29 answers

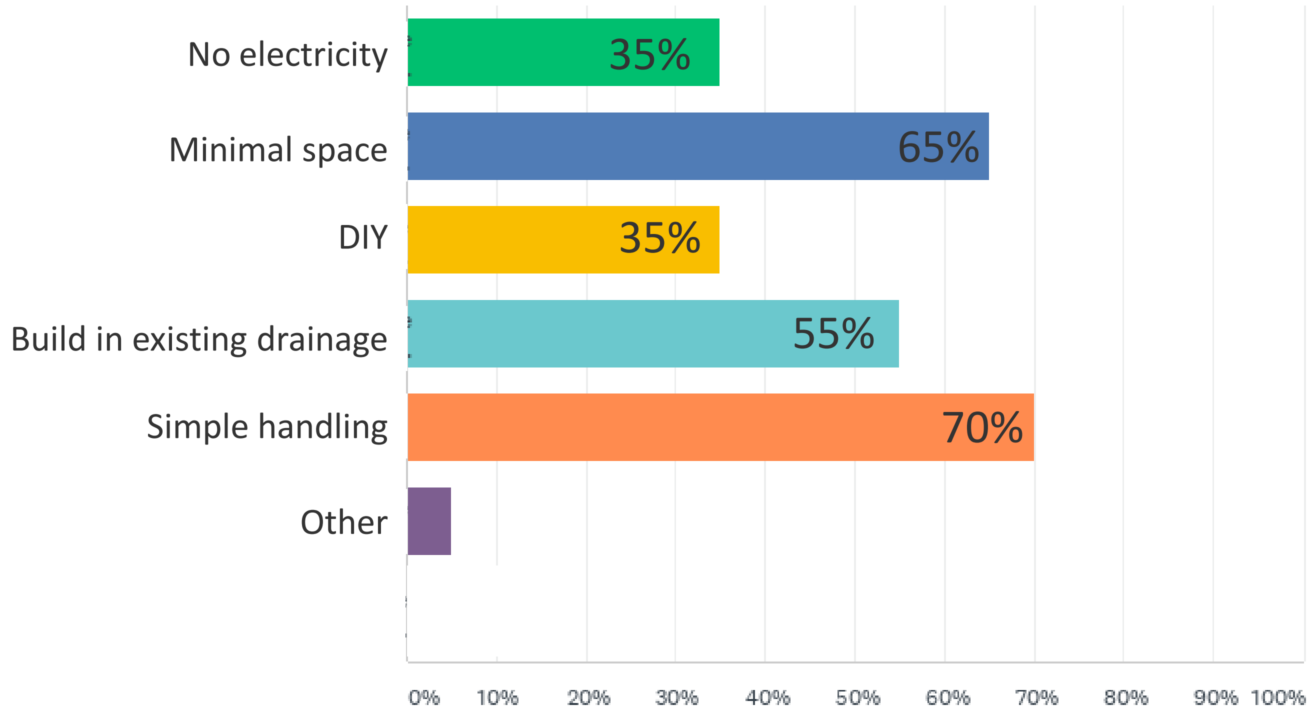
Are you familiar with end-of-pipe technology to remove nutrients from agricultural waters?

Beantwoord: 29   Overgeslagen: 0



Preferential requirements for the filter are:

Beantwoord: 20   Overgeslagen: 9



Which investment cost is acceptable?

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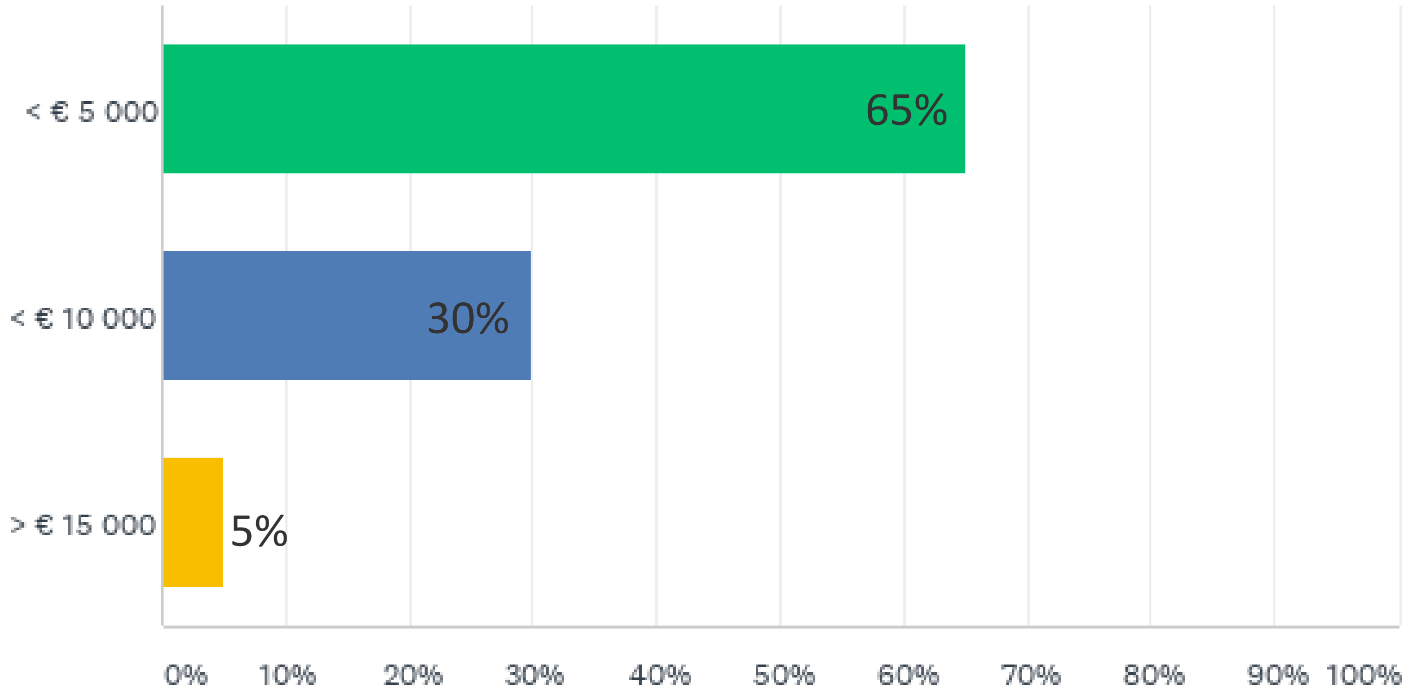

Within which time frame would you consider this investment?

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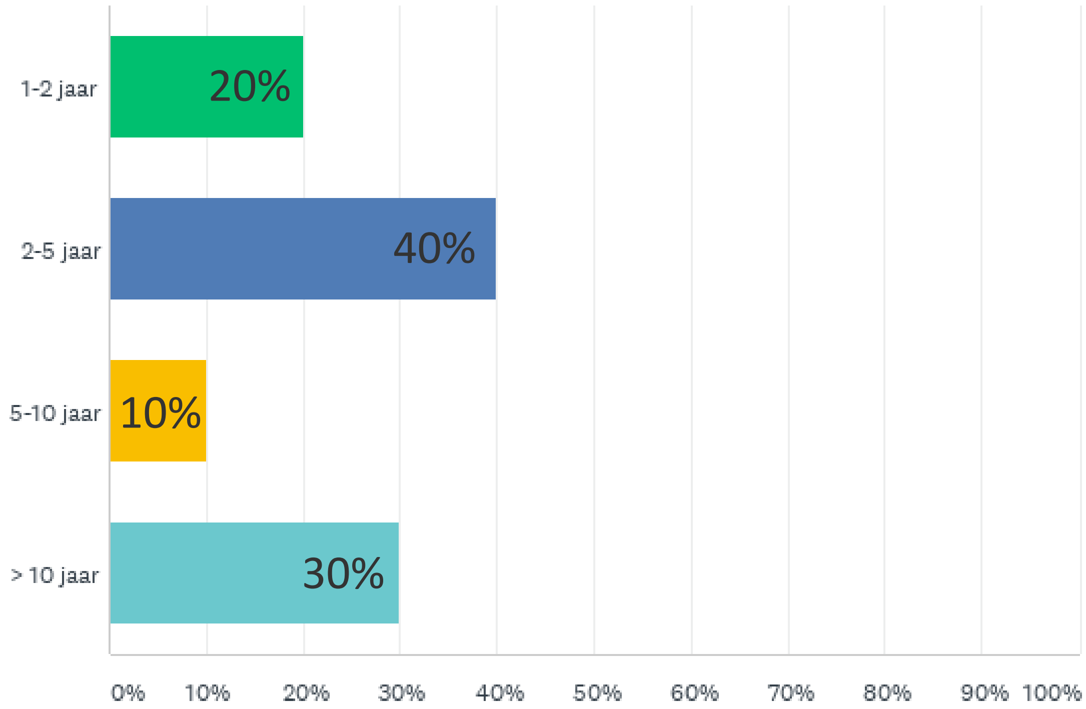
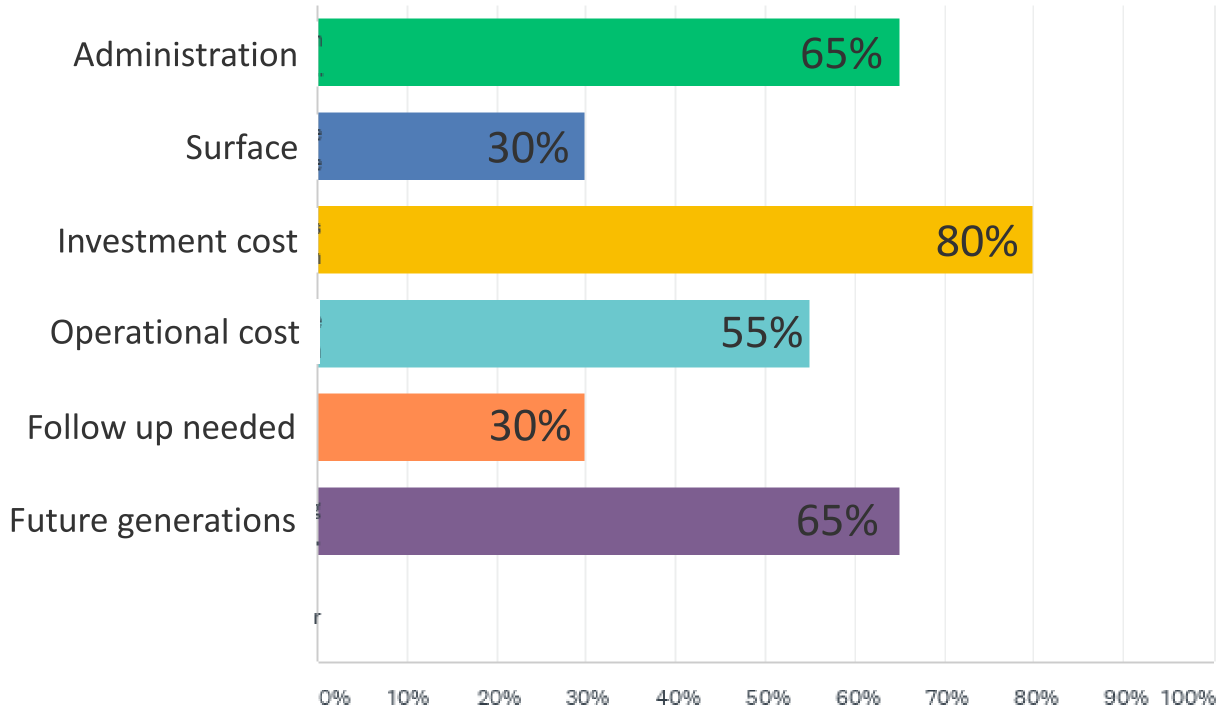


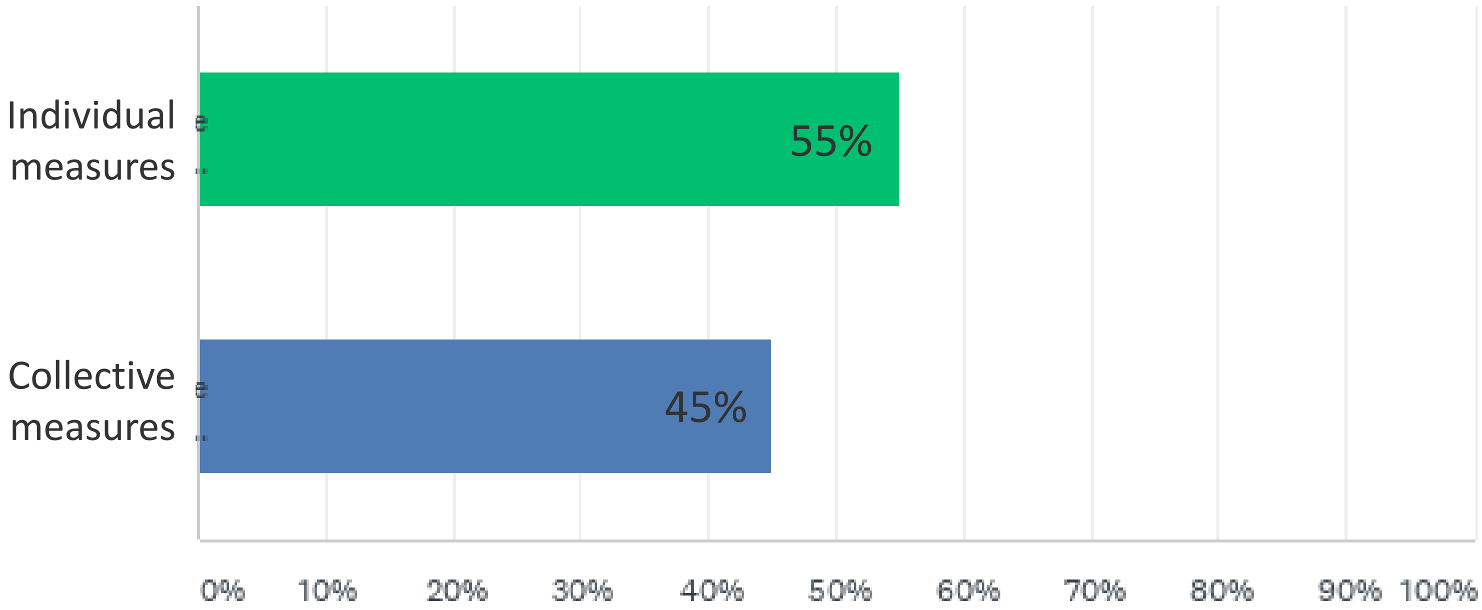
Which factors influence your choice for a certain technology?

Beantwoord: 20   Overgeslagen: 9



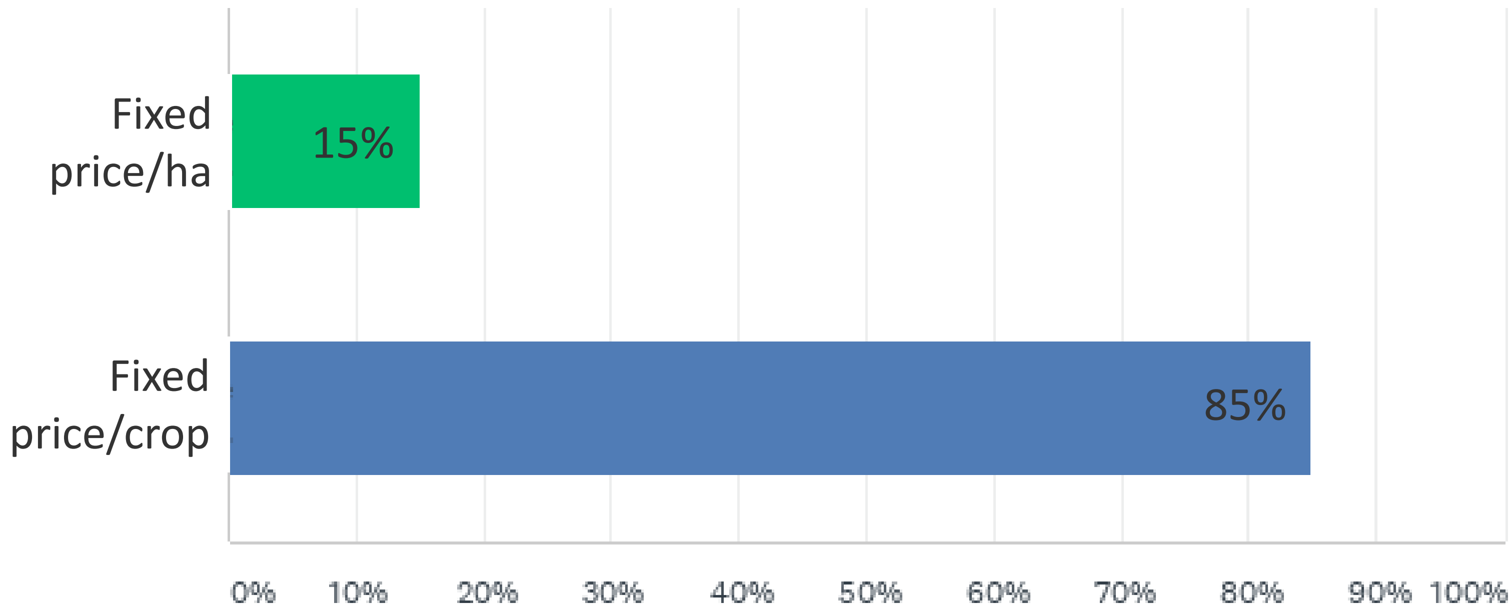
I prefer:

Beantwoord: 20 Overgeslagen: 9



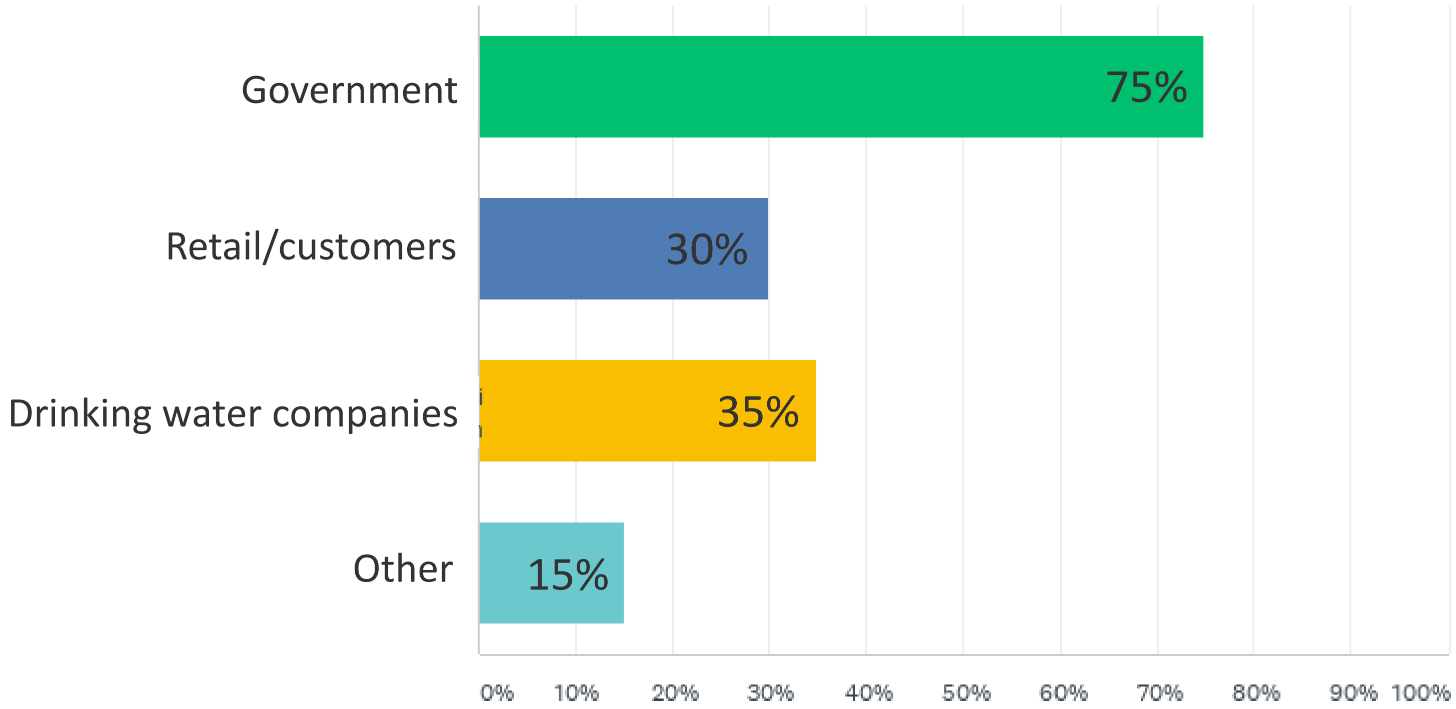
In case of collective measures, which financing system is preferential?

Beantwoord: 20   Overgeslagen: 9



In case of collective measures, who else should pay?

Beantwoord: 20   Overgeslagen: 9



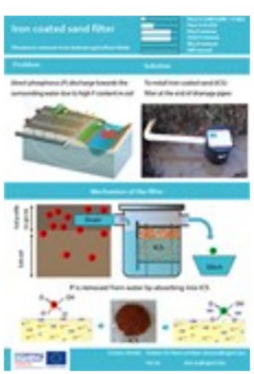
Farmers' opinion

- Simple technology required minimum of space
- Cost < € 5000
- Investments within 2-5 years
- Individual measures <-> collective measures
- Fixed price/crop

Nuredrain information

- [NuReDrain, Interreg VB North Sea Region Programme]
- Scientific articles
- Filter fact sheets
- Videos
- MBBR manual: working principle, calculation tool, DIY build instruction

Filter Fact Sheets



Filter Construction Manuals

Field visits with sun

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Field visits with rain

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Field tests in summer

Field tests in winter

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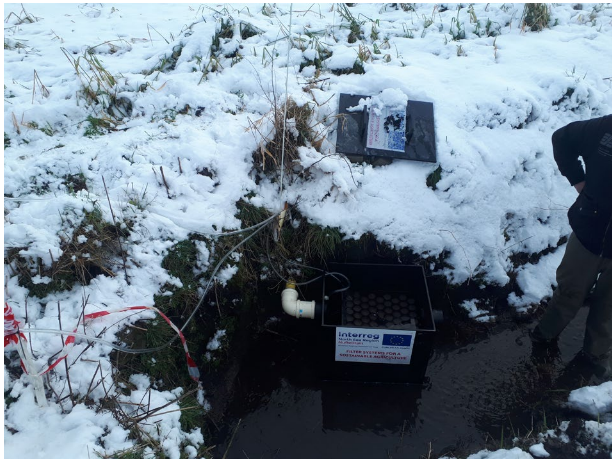

Acknowledgements