Valorisation of P-saturated filter materials

Els Pauwels – PCS Ornamental Plant Research
Nico Lambert – KU Leuven
Process & Environmental Technology Lab

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P-saturated filter materials

2 types of valorization strategies:

- P desorption and recovery: Desorption capacity → P-desorption tests on lab scale
- Using filter material as fertilizer: Fertilizing capacity → pot and field trials

Economic study:

- For which materials is it economically viable to perform P desorption?
- Evaluation of the specifications (quantity, quality) requested by fertilizer production companies to check if the demands can be met?

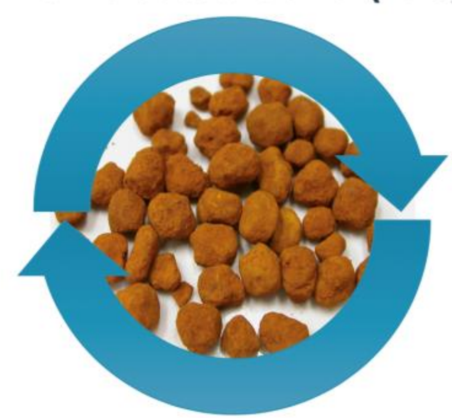
ICS

- P-saturated ICS available in relatively large amounts
- ICS (Iron coated sand) as a reference material:
  - Waste product from drinking water production
  - Good removal of P - rich drainage waters
  - High hydraulic conductivity (depending on size of particles)
  - (Sufficiently) available and (relatively) cheap

First valorization strategy:

- The main objectives: **Regeneration of the saturated sorbents** and **recovery of phosphorus**.
- A desorption process using an **alkaline** solution is proposed without harming the sorbent granules.

Iron Coated Sand (ICS)



Integration of P-adsorbing material in a circular process

Concept of ad/desorption

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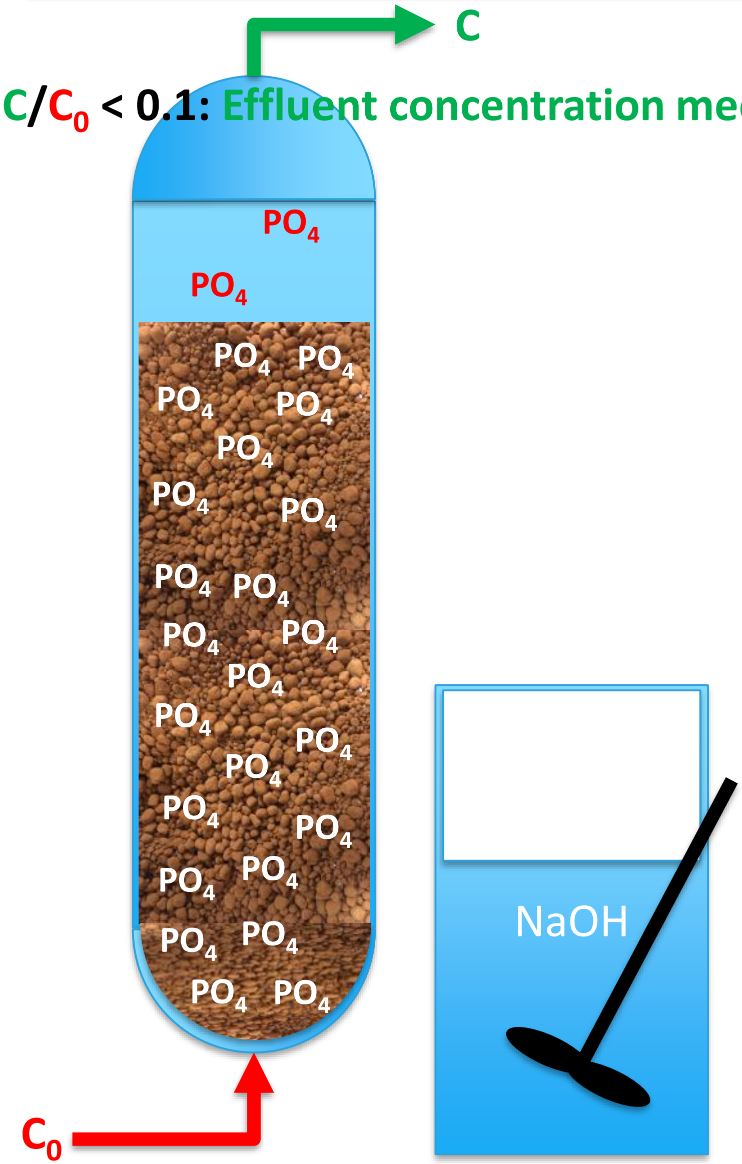
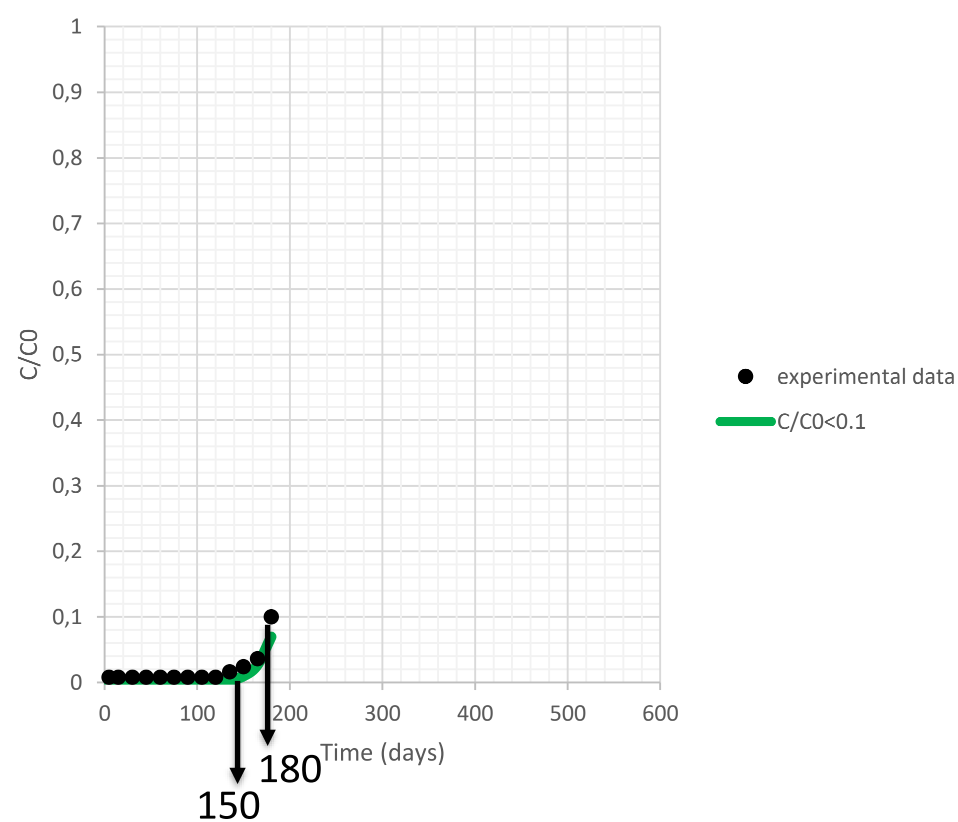
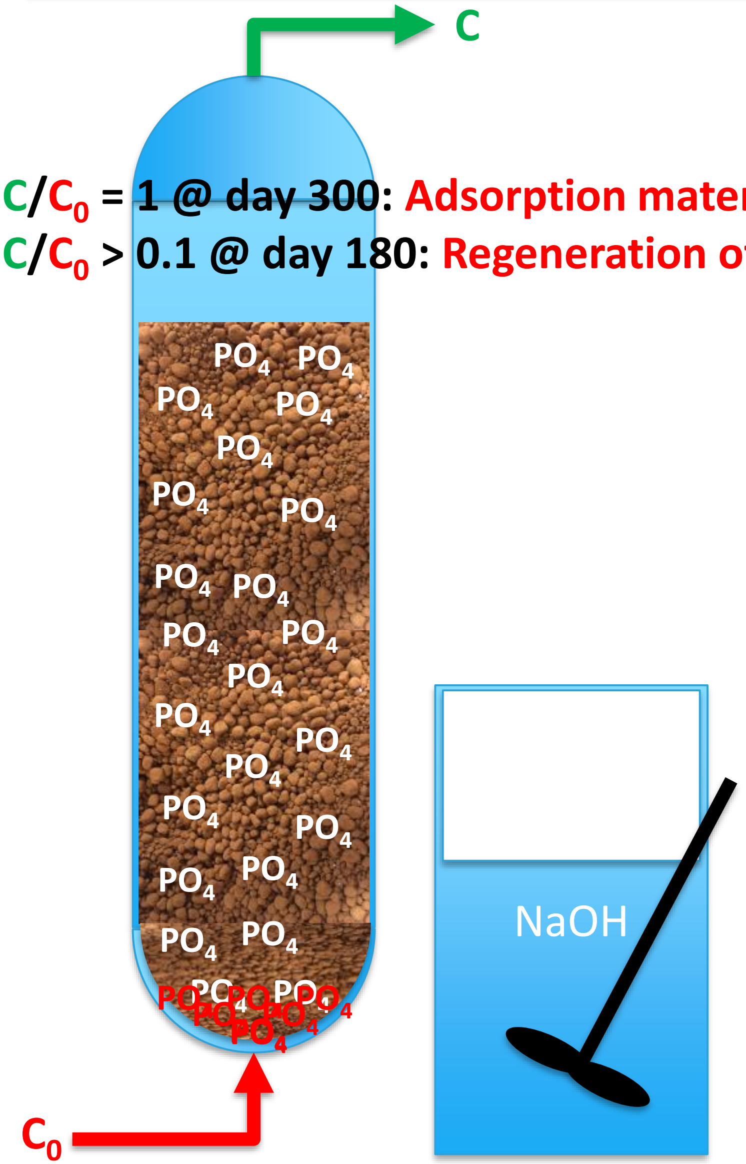
$C/C_0 < 0.1$: Effluent concentration meets the discharge limit



Adsorption Phase: Day 0-180



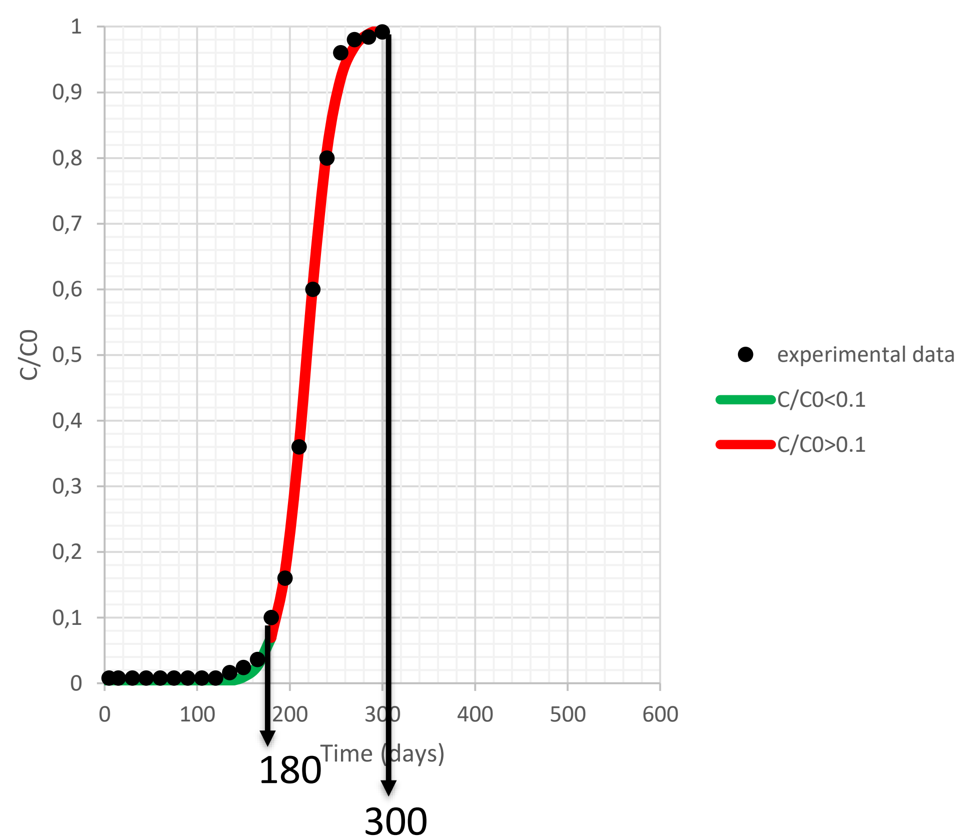
Concept of ad/desorption



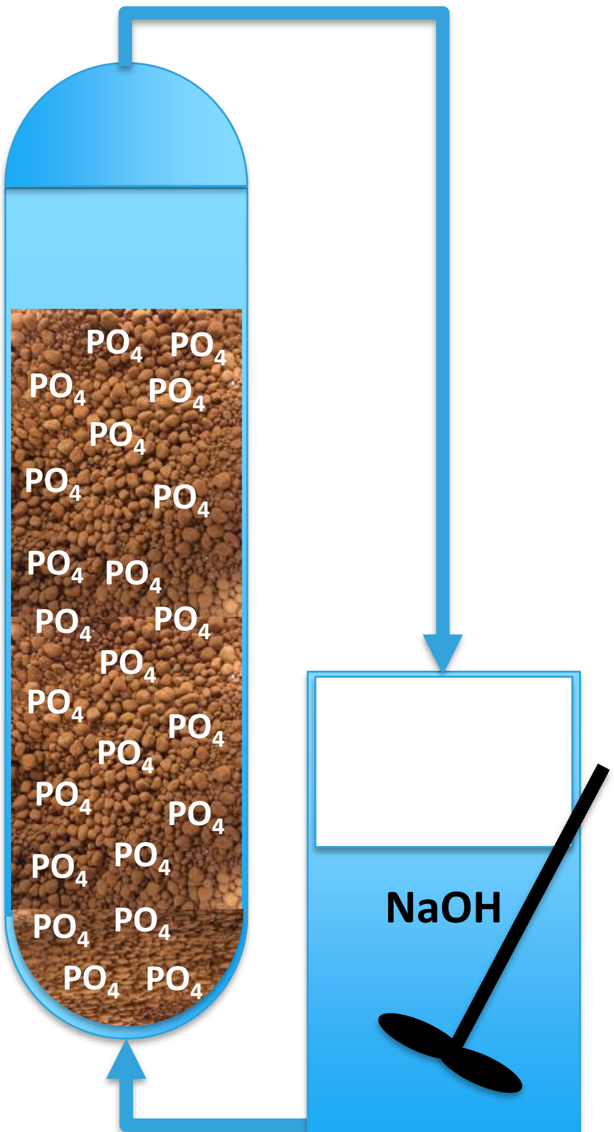
$C/C_0 = 1$ @ day 300: Adsorption material (ICS) is completely saturated

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

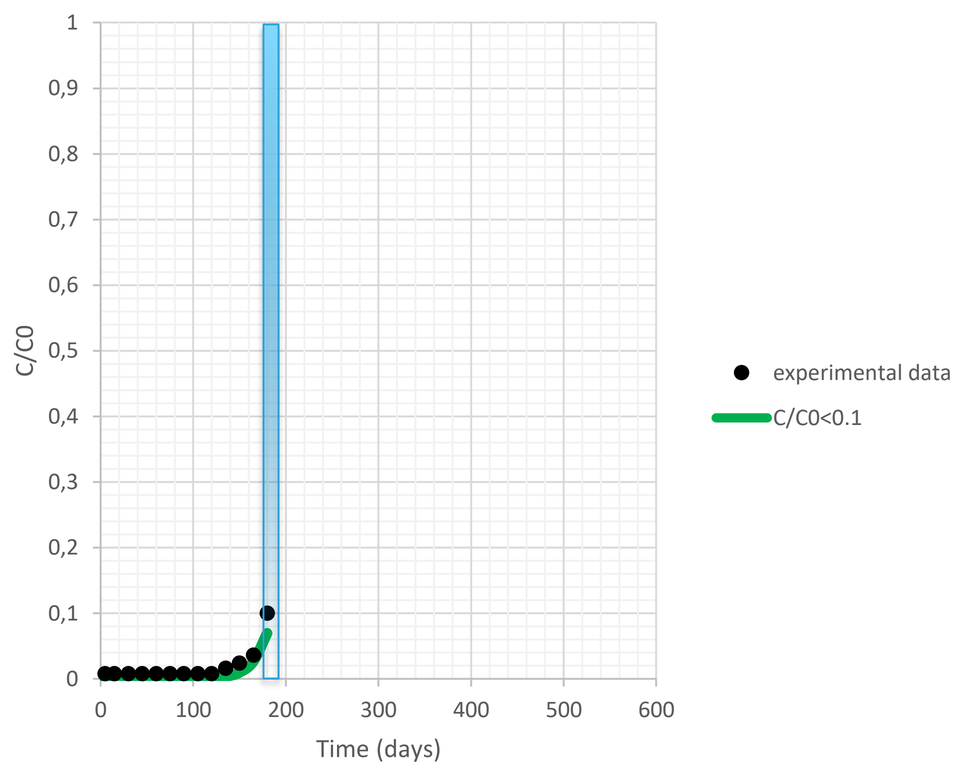
Adsorption Phase: Day 180-300



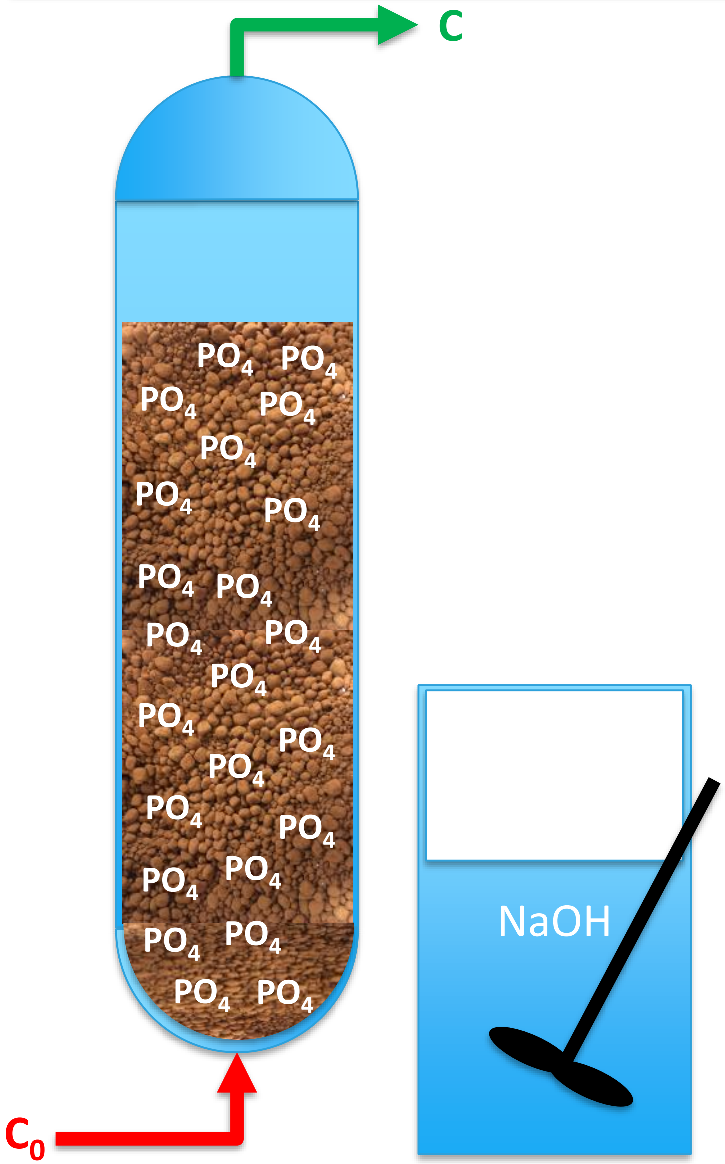
Concept of ad/desorption



Desorption Phase: Day 180



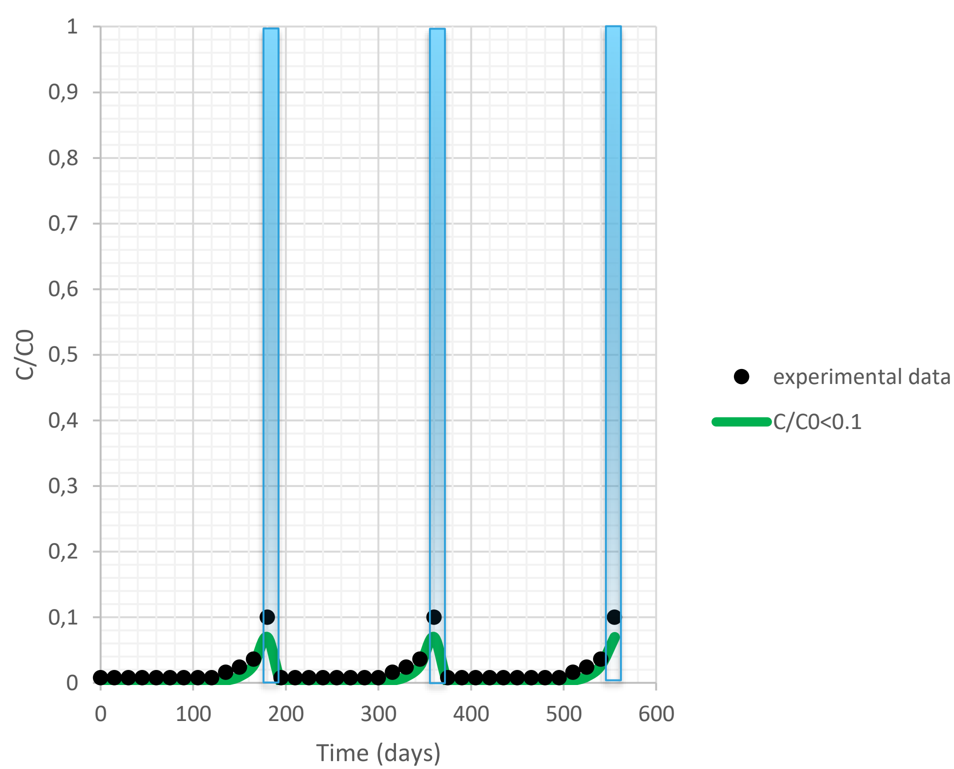
Concept of ad/desorption



Objectives:

- **Extend filter breakthrough time**
- **How many times?**

Intermittent regeneration of ICS



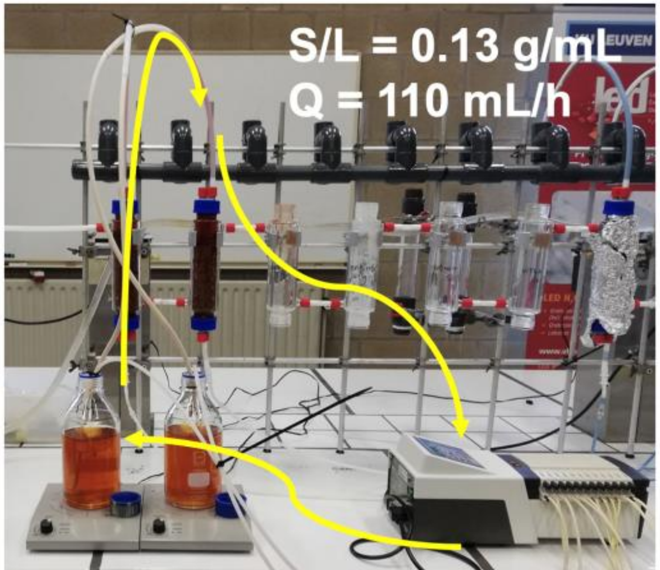
Materials & Methods

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

   <u>Variable parameters:</u>
   - 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



Continuous filter desorption experiment

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.

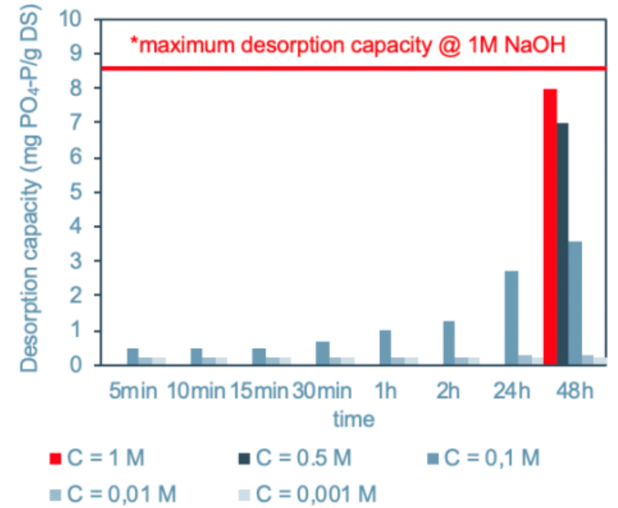


Figure 1: Influence of NaOH concentration and desorption time.

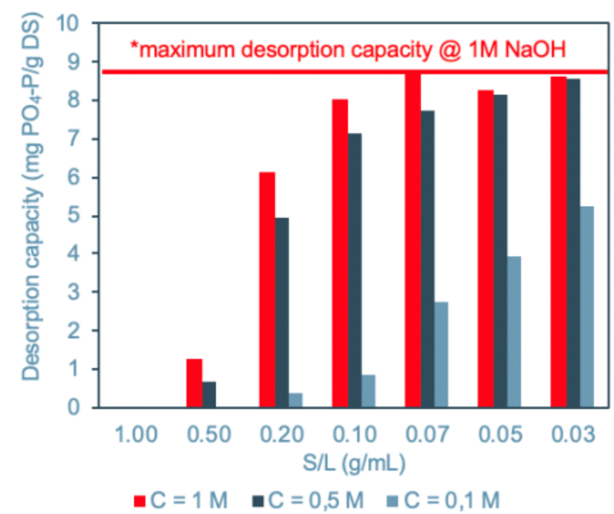


Figure 2: Influence of solid/liquid ratio.

Results & Discussion
Continious filter experiments

- **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.

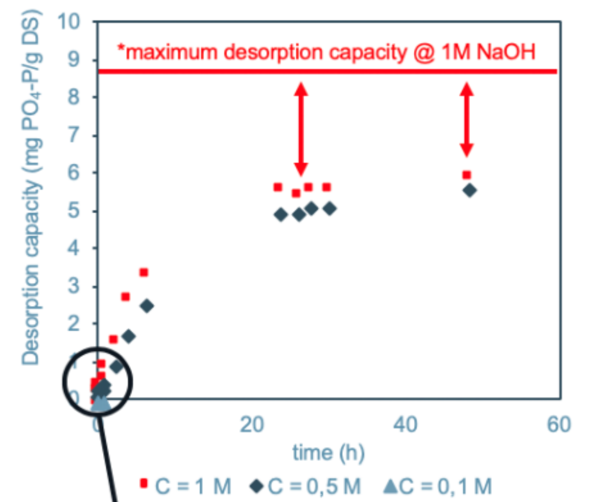


Figure 3: Continuous filter desorption experiment and the effect of the NaOH concentration on desorption capacity

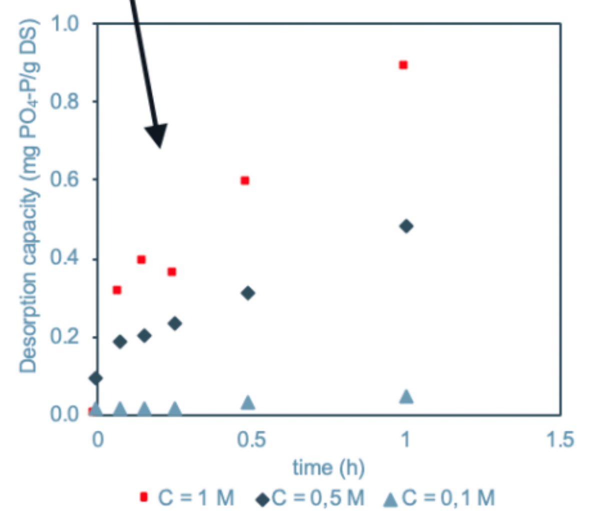


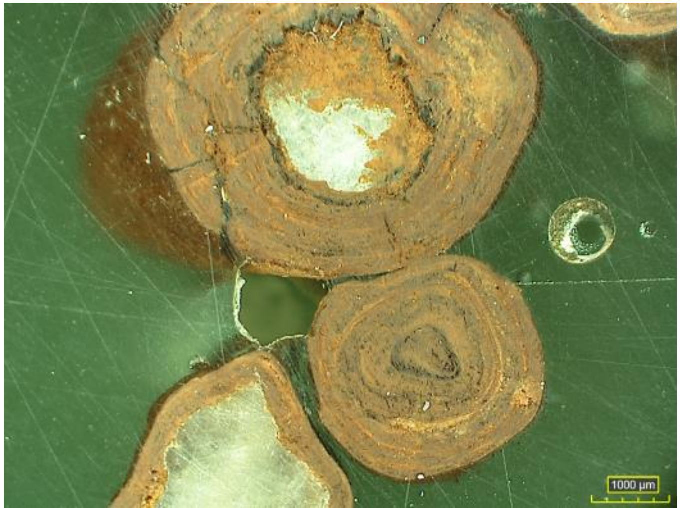
Figure 4: The progress of the desorption during the first hour of the continuous filter desorption experiment

Results & Discussion
SEM-EDX analysis

- Energy-dispersive X-ray (EDX) Analysis with a Scanning Electron Microscope (SEM)

- **Figure 5:** The ICS granules have a solid structure with a sand core surrounded with iron. The phosphorous is accumulated at the sand core of the granule.

polished ICS granules embedded in a resin



Si – Fe – P analysis by EDX

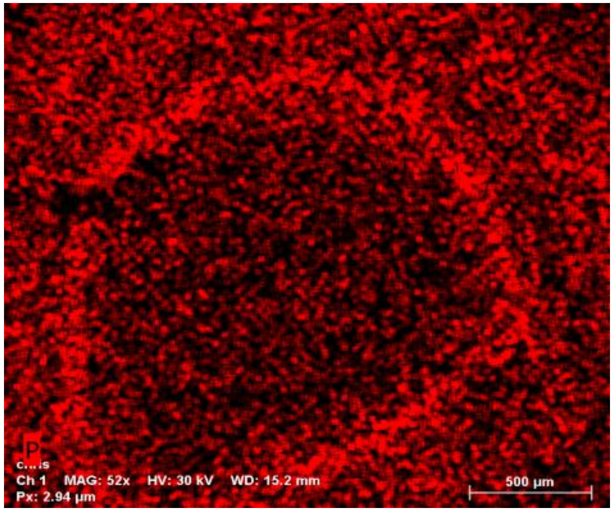


Figure 5: SEM-EDX analysis of the ICS granules

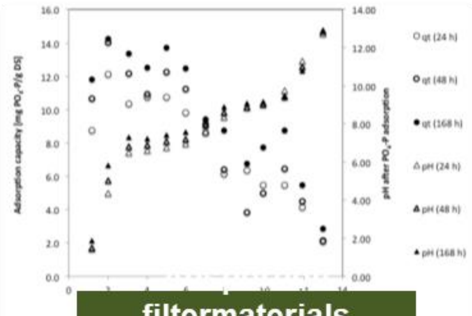
Conclusions

- 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

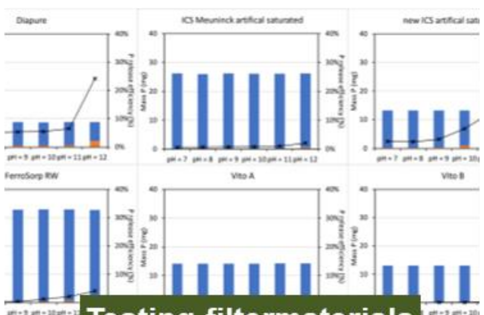
Other methods for desorption of P?

P – saturated ICS

- Use saturated ICS as a P – fertilizer?
- Strong Fe - P_2O_5 – adsorption
- Release P_2O_5 by adding Phosphate Solubilizing bacteria (PSB's)?
- Aqua – regia extraction ICS : 20,935 g P/kg DM
- P = non-mobile element : Include effect of fertilizer placement (broadcast vs point application)



filtermaterials



Testing filtermaterials

Test on azalea

Pot trial 2017:

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

Trial PCS 2018: Buxus, Lavendula and Hedera

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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 was 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

Trial PCS 2018: Buxus, Lavendula and Hedera

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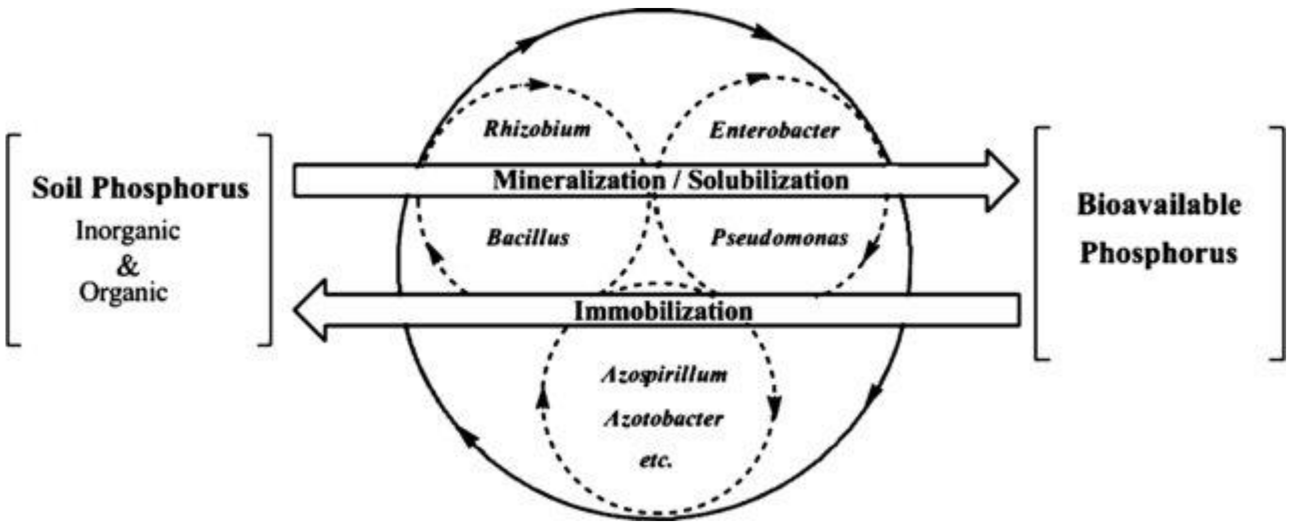
Standard N + standard P	Standard N without P	Standard N without P + 30% ICS

PSB

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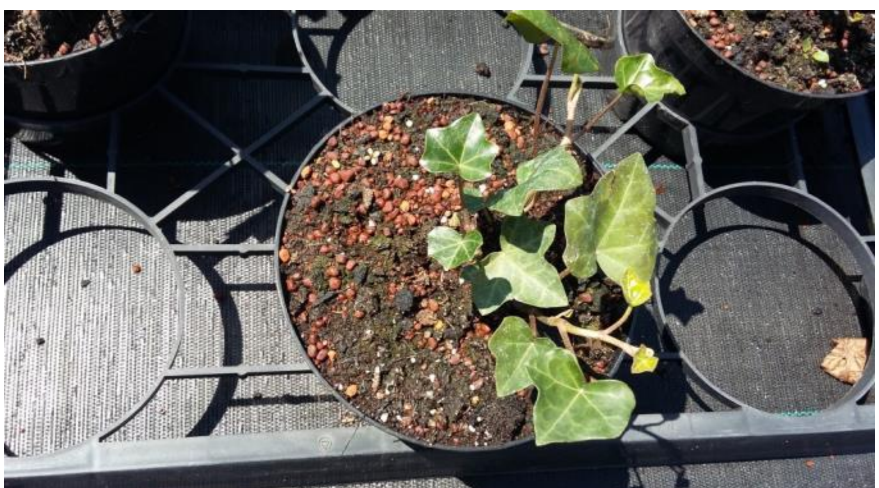
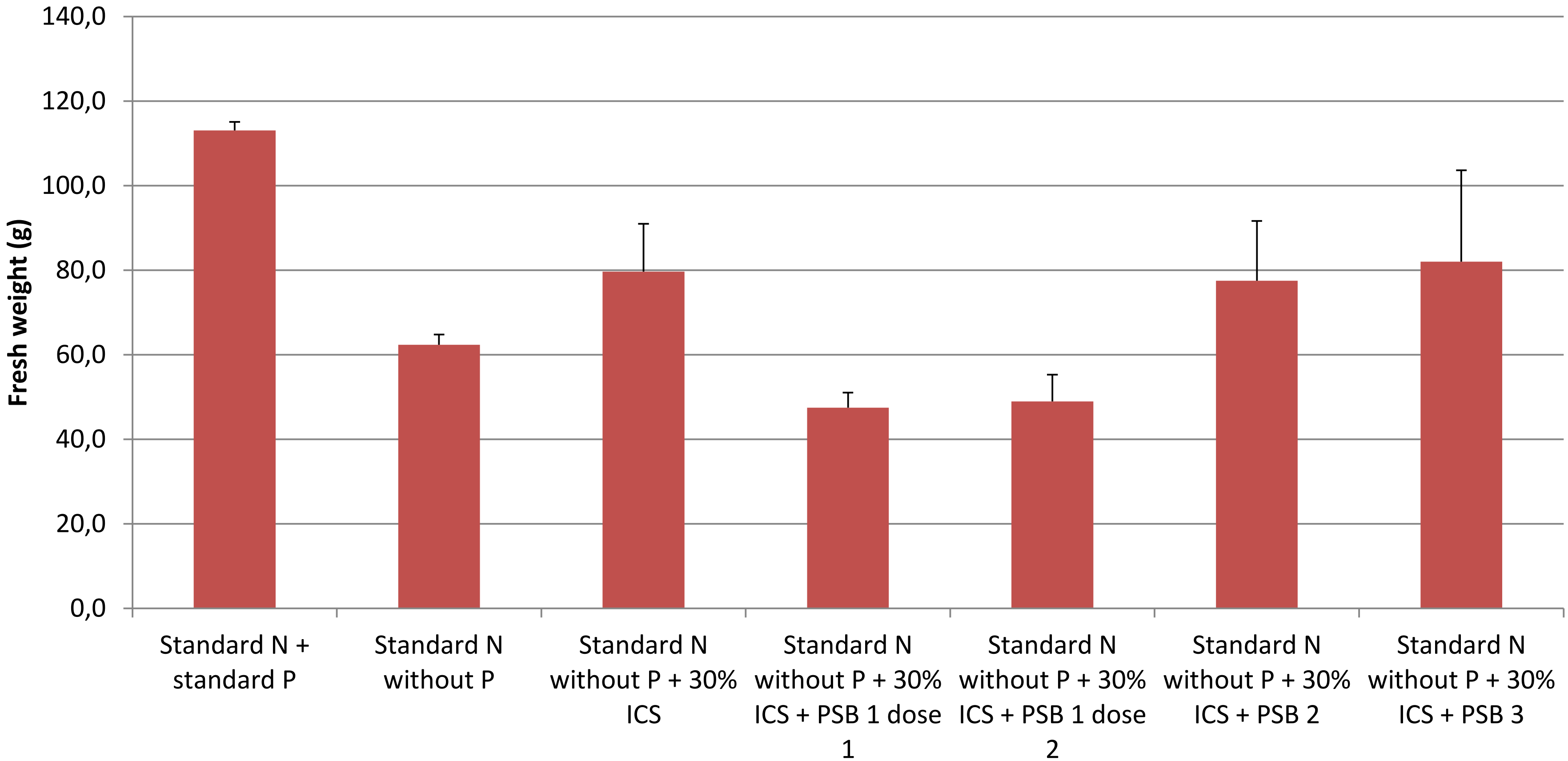
Treatment	
1	Standard N and Standard P
2	Standard N without P
3	Standard N without P but with 30% ICS granules
4	Standard N without P but with 30% ICS granules + dose 1 of PSM1
5	Standard N without P but with 30% ICS granules + dose 2 of PSM1
6	Standard N without P but with 30% ICS granules + dose 1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

Trial PCS 2019: Hedera



Trial PCS 2019: Hedera



Trials at Inagro

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Spring 2019 :
- growth chamber experiment in endive :
- proof of concept
  - Use of ICS as a P – fertilizer
  - Use of PSB's

Summer 2019 :
- Pot experiment in maize (outdoors)
- Evaluation of commercial products

Autumn 2019 (on going):
- Pot experiment in endive
- Additives for PSB's
- 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

- Performed in open air
- In pots of 7 l 4 maize seeds were sown
- Per plot 4 pots were provided
- These 4 pots were grouped in a container
- The trial was performed in 4 replicates
- Interim harvest (3 plants per plot) for analysis of nutrient content during the youth growth stage

Pot trial maize P-fertilisation with ICS

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All treatments received an equal start doses of nitrogen, potassium and phosphate based on fertilization advice (sowing date 13/05/2019)

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

Pot trial maize P-fertilisation with ICS

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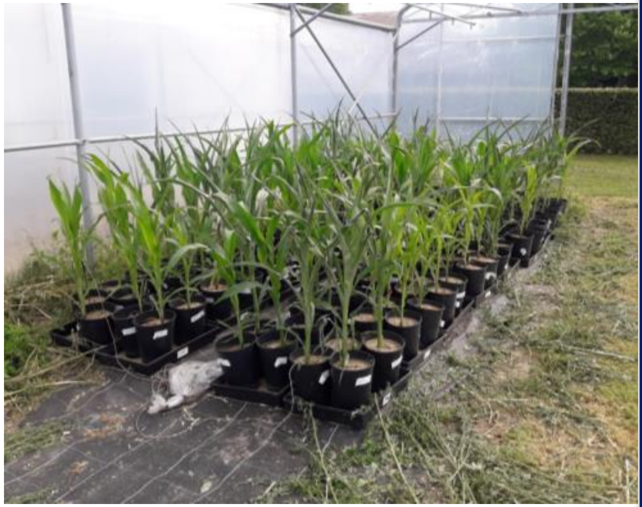
Nutrient content at interim harvest (11/06/2019)

Nr	Treatment	Tot. nitrogen (g/kg DM)		Potassium (g/kg DM)		Phosphor (g/kg DM)	
1	Control	50,70	ab	31,10	bc	2,43	c
2	APP	53,89	a	32,66	ab	5,36	a
3	TSP	48,50	b	34,84	a	4,45	b
4	Ecostyle PT mix + ICS	48,47	b	31,84	bc	2,37	c
5	Ecostyle PT mix	48,47	b	31,76	bc	2,42	c
6	Ecostyle PT mix + TSP	49,85	ab	33,97	ab	4,23	b
7	P putida + ICS	51,45	ab	29,02	c	2,24	c
8	P putida	51,24	ab	31,08	bc	2,33	c
9	P putida + TSP	52,79	ab	35,42	a	4,46	b



Pot trial maize P-fertilisation with ICS

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Nr	Treatment	Length (cm)		DM yield (%)	
1	Control	163,75	cd	84,22	b
2	APP	192,50	ab	130,23	a
3	TSP	195,50	a	116,54	a
4	Ecostyle PT mix + ICS	169,25	bcd	84,53	b
5	Ecostyle PT mix	165,25	cd	84,59	b
6	Ecostyle PT mix + TSP	197,00	a	136,83	a
7	P putida + ICS	159,00	d	57,74	b
8	P putida	159,00	d	69,74	b
9	P putida + TSP	187,75	abc	135,58	a

-> 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

Pot trial endive (2018-2019)

P fertilization value of phosphorous saturated ICS combined with P solubilizing bacteria (PSB) in low phosphorous content soil on a high responsive crop (= endive)

- Trial in growth chamber (2018)
- Pot experiment (2019 – on going)

Pot trial endive (2018 -2019)

Overall conclusion pot trials endive

- No indication that phosphorus rich material (ICS) has a potential as P-fertilizer
- Use of ICS at the root zone (planting hole) even worse vs mixed use (mixed with soil)
- No added value of PSB's in combination with ICS

Other possibilities to use ICS?

- As an addition to the substrate?

Trial PCS 2018: ICS as substrate?

Chlorophytum

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

left without ICS – right with ICS

Trial PCS 2018: 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

Trial PCS 2019: 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 2019: 14 different plant species

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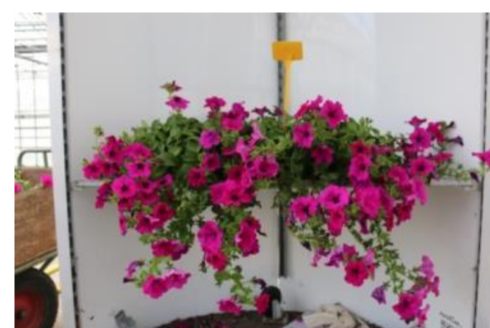
Trial PCS 2019: 14 different plant species



Other possibilities to use ICS?

- As an addition to the substrate?
- As a cover material on the pots

Trial PCS 2019: As a cover material

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Trial PCS 2019: As a cover material

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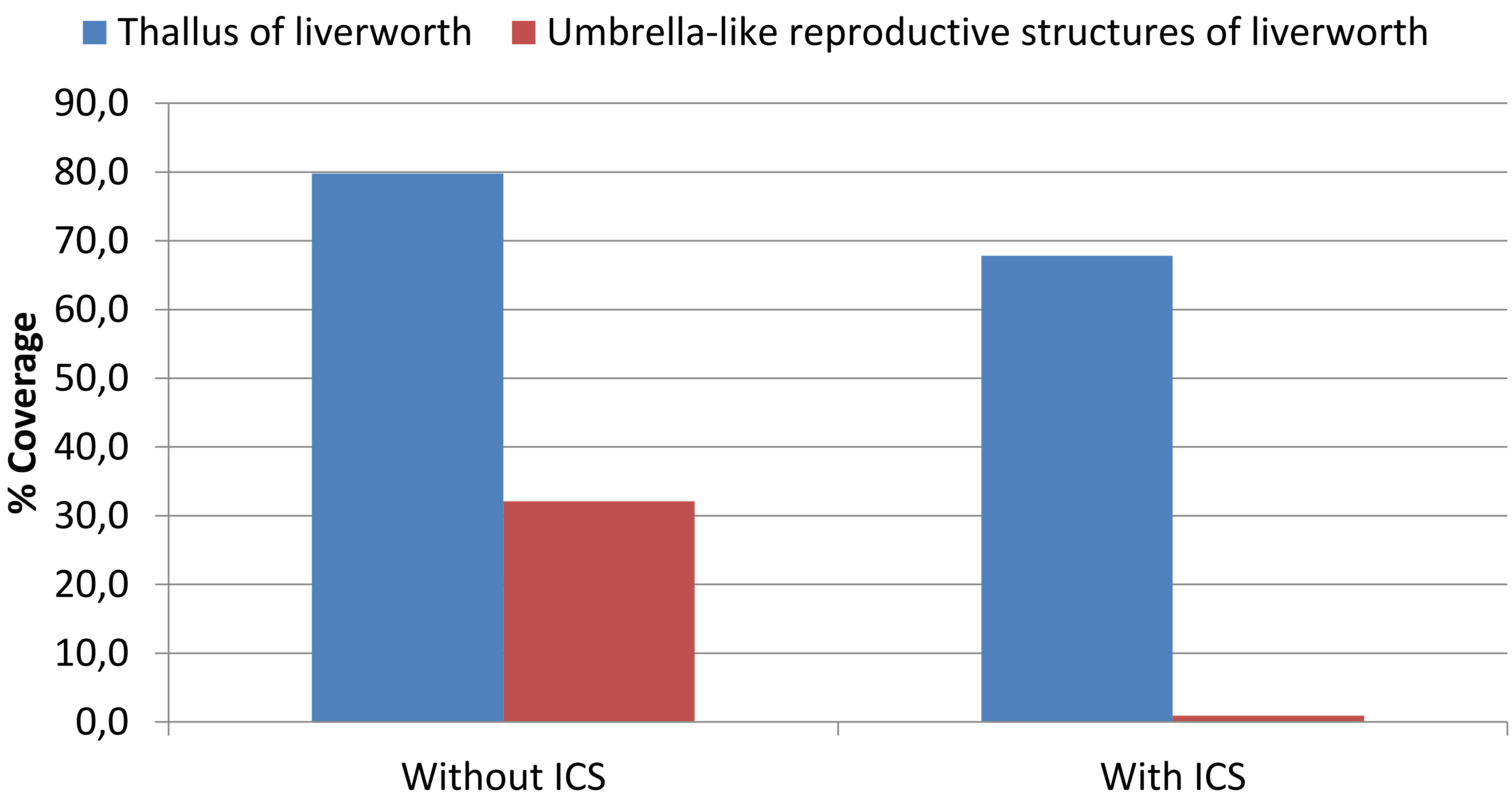
Camellia



Other possibilities to use ICS?

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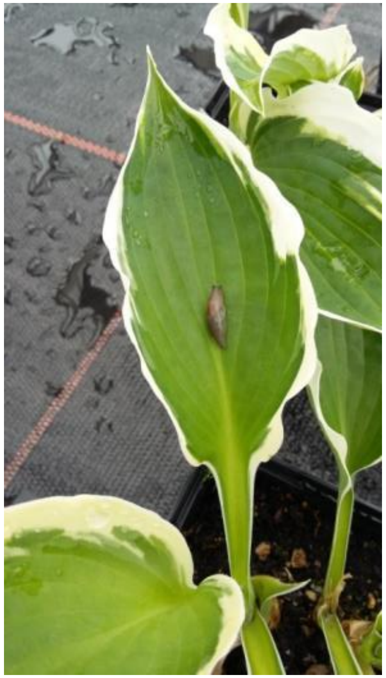
- As an addition to the substrate?
- As a cover material on the pots
- Against slugs?
- Others?

Other possibilities to use ICS?



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

Q & A