

Recovery of Phosphorus From Dried Sewage Sludge for Fertilizer Formulations

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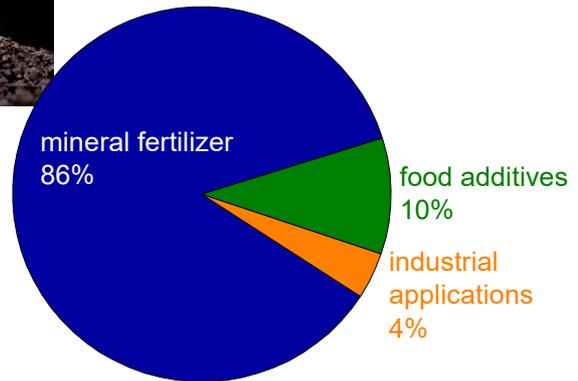
agenda

- motivation
- PULSE process and pilot
- solid-liquid-liquid equilibrium tool
- results: pilot trials and simulation
- summary

phosphorus background



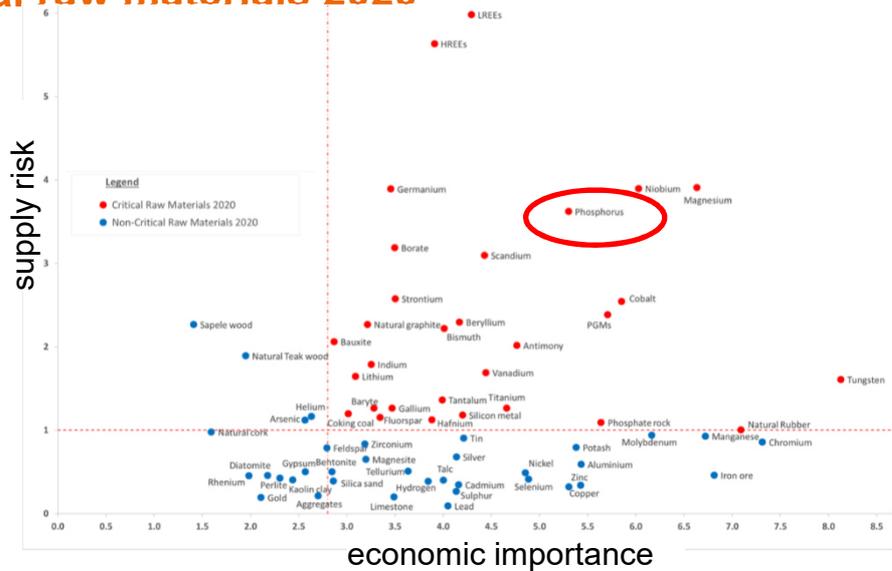
rock phosphate



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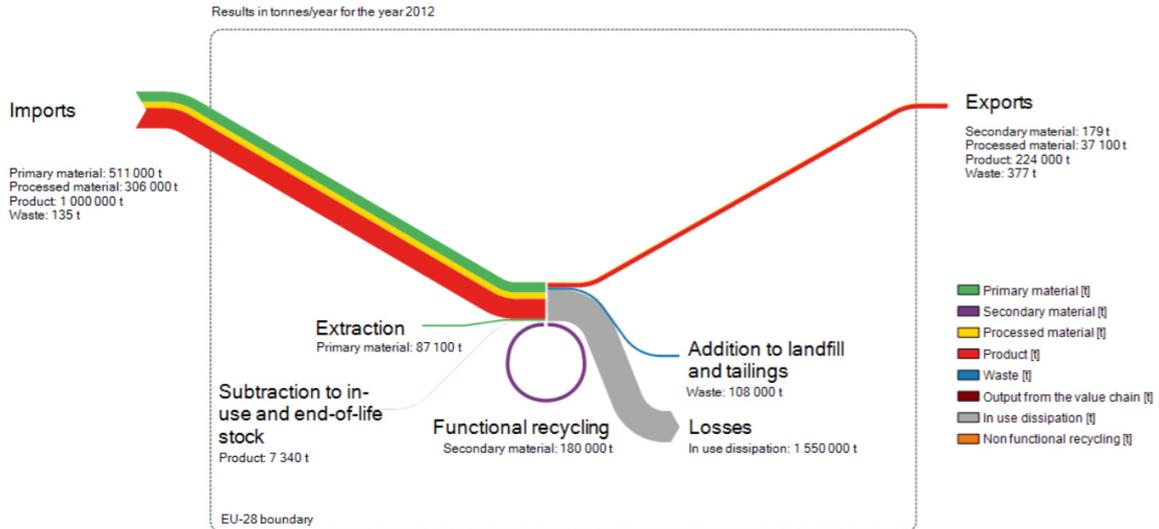
EU critical raw materials 2020



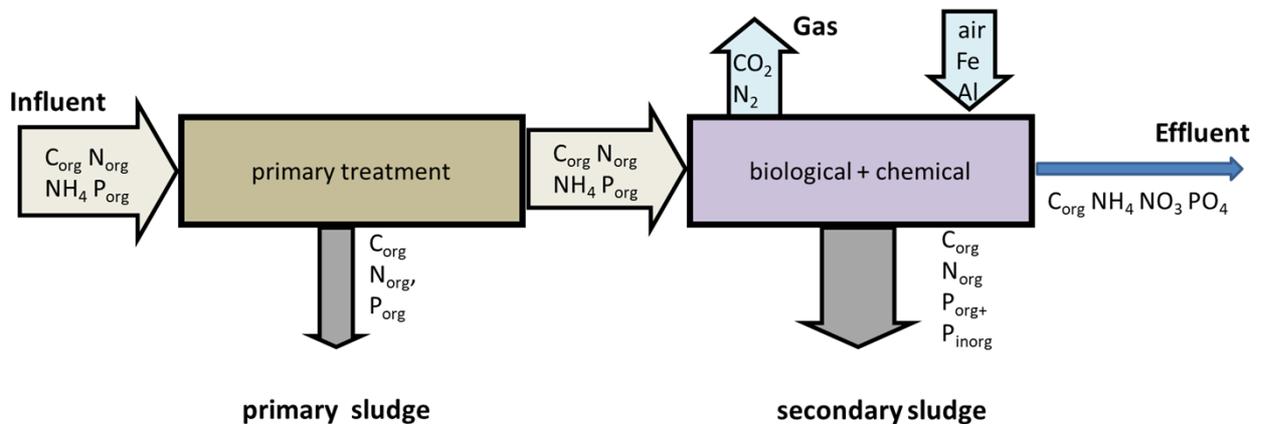
4



P-flows in EU 2012



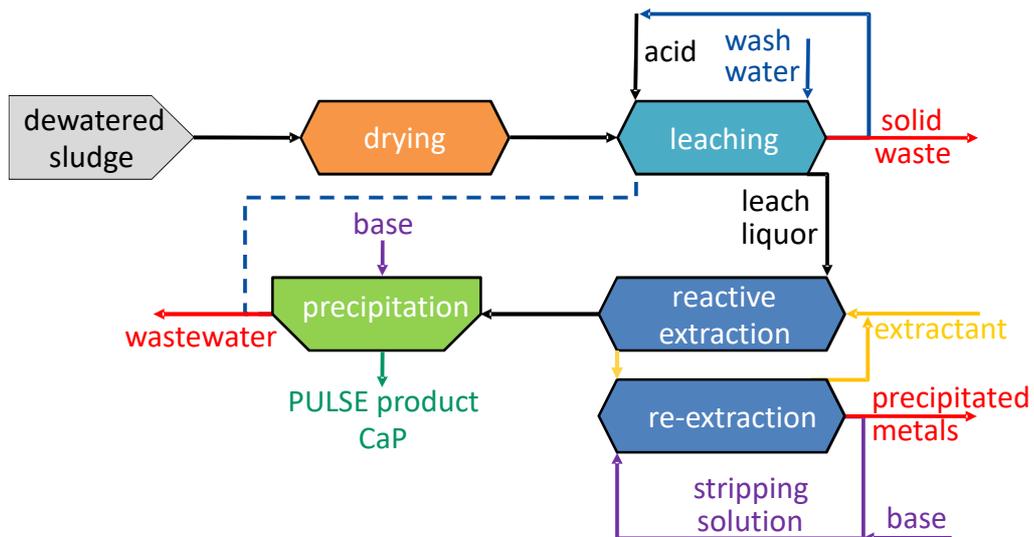
P removal in wwtp



Phos4You EU project demonstrators

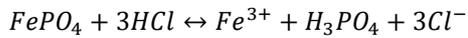
demonstrator	country	source	process
EuPhoRe GmbH	Germany	(digested) sludge	pyrolysis followed by incineration
Lippeverband with REMONDIS Aqua	Germany	sewage sludge ashes	chemical leaching followed by ion-exchange
PULSE process, ULiège	Belgium	dried sludge	chemical leaching, reactive extraction, precipitation
IRSTEA and Véolia	France	sewage sludge	bio-acidification, ion-exchange, precipitation
Véolia with CIT and GCU	France	wastewater	precipitation
GCU	Scotland	wastewater	P uptake by algae
ERI and Véolia	Scotland	wastewater	adsorption of P

PULSE (Phosphorus University of Liege Sludge Extraction)



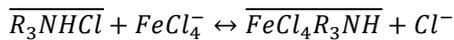
reactions in PULSE process (examples)

- leaching of P and metals from sludge



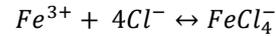
$$K_l = \frac{c_{Fe^{3+}} c_{H_3PO_4} c_{Cl^-}^3}{c_{FePO_4} c_{HCl}^3}$$

- reactive extraction of metals with organic extractant



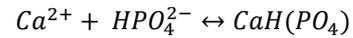
$$K_e = \frac{c_{\overline{FeCl_4 R_3NH}} c_{Cl^-}}{c_{R_3NHCl} c_{FeCl_4^-}}$$

- speciation in aqueous solution



$$K_a = \frac{c_{FeCl_4^-}}{c_{Fe^{3+}} c_{Cl^-}^4}$$

- precipitation of CaP



$$K_p = \frac{1}{c_{Ca^{2+}} c_{HPO_4^{2-}}}$$

Solid-Liquid-Liquid Equilibrium tool

- charge balance

$$0 = \sum_{i=0}^n c_i Z_i$$

- mass balances

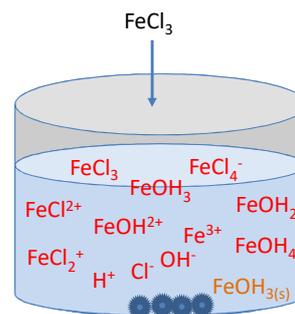
$$c_{tot,j} = \sum_{i=0}^n \nu_{i,j} c_i$$

- law of mass action

$$\log K_m = \sum_{i=0}^n \nu_{i,r} \log a_i$$

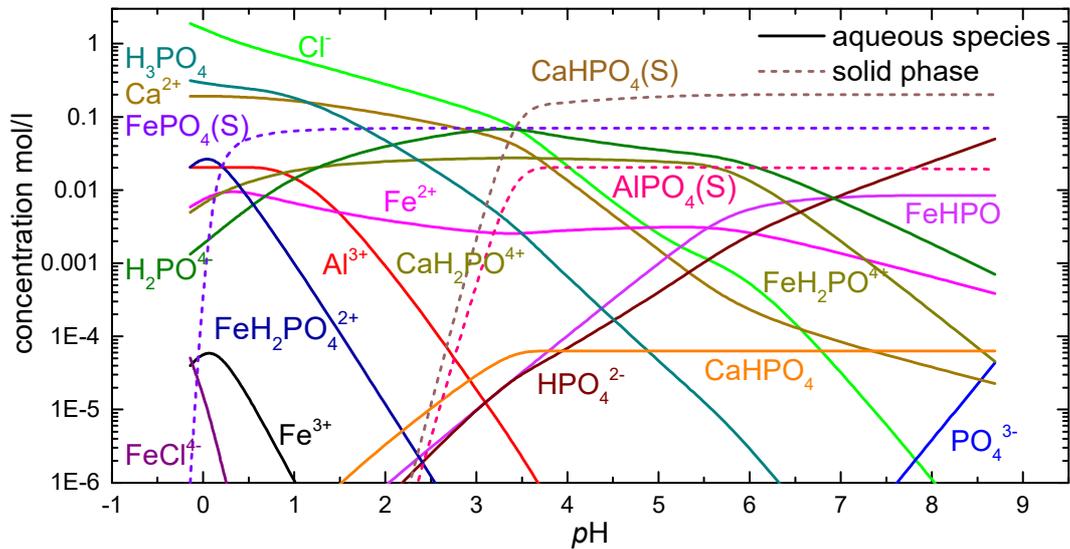
- Ion Activity Product: $IAP = a_A^{\nu_1} a_B^{\nu_2}$

- Saturation Index: $SI = \log IAP - \log K_{sp}$

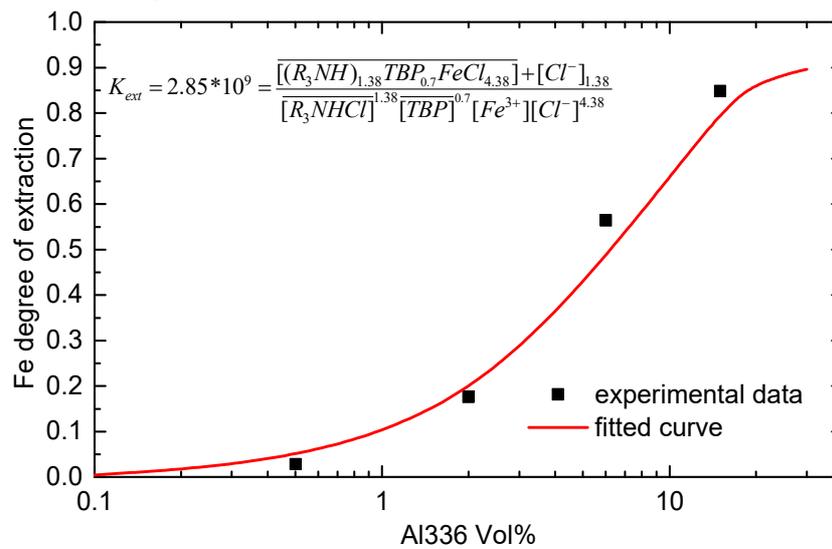


$a_i = \gamma_i c_i$ (activity)
 γ_i = activity coefficient
 c_i = concentration
 i = species
 j = master species

SLLE tool simulation



parameter fitting with SLLE tool



sludge drying

dewatered sludge (20% to 22% DM)



- biologically unstable \Rightarrow storage & transportation issues
- pasty \Rightarrow requires 2 to 3 times more water & acid for leaching (0.06€/mol acid)
- difficult to filter at low pH

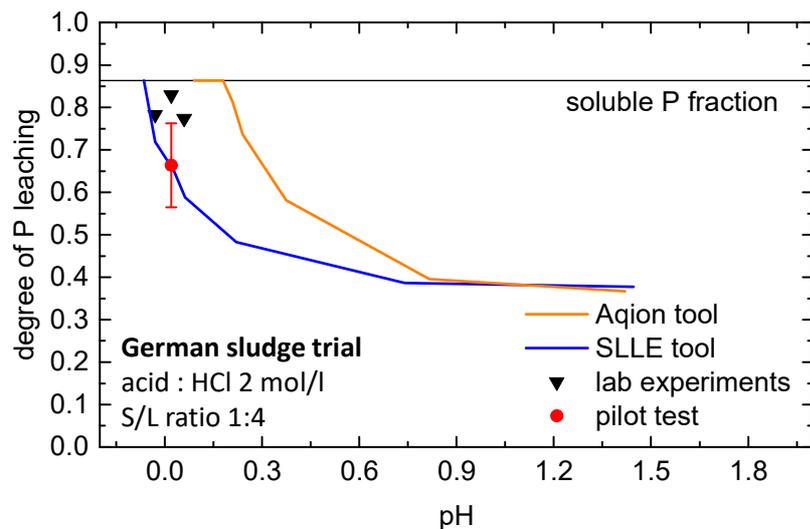
dried sludge (>95% DM)



- stable solid \Rightarrow easier storage & transportation
- mineral like \Rightarrow less water & acid for required for leaching (drying cost = 0.1 – 0.21 €/kg dewatered sludge)
- easier to filter



phosphorus leaching from sludge



PULSE demonstrator



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15

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PULSE demonstrator

- mobile unit
- capacity to treat up to 80 kg of dewatered sludge per batch
- 5 horizontal mixer-settlers
- locations:
ULiège
Oupeye WWTP
Bo'ness, Scotland



©uliege-michel houet

PEPs
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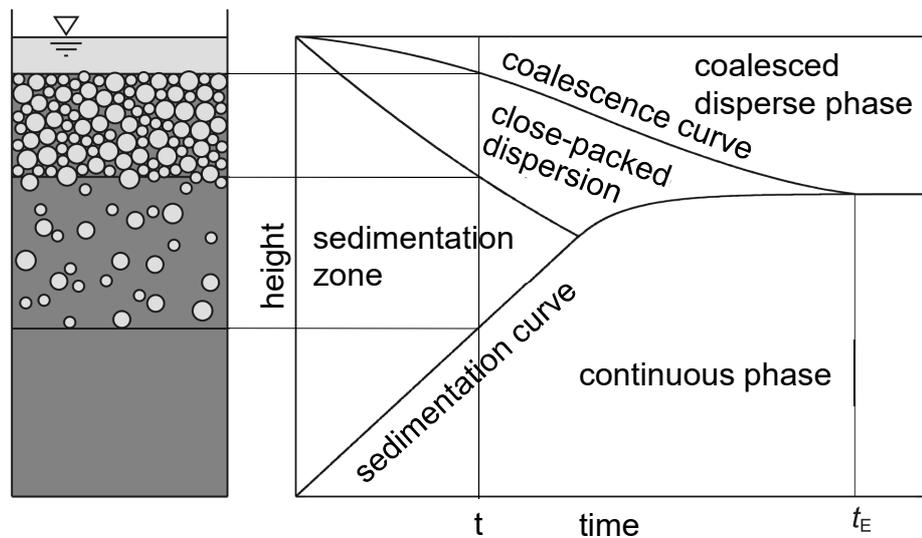
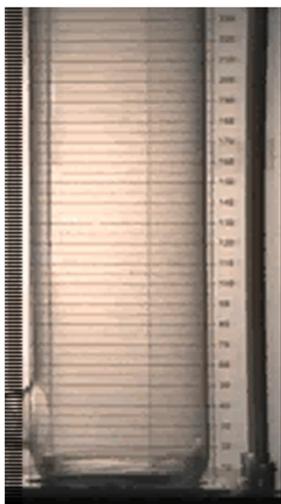
16

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standardized settling cell



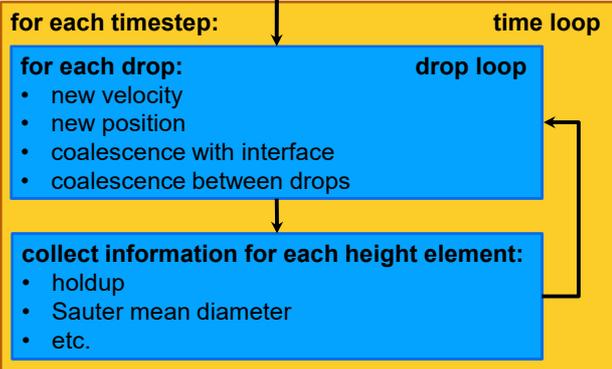
general settler design concept



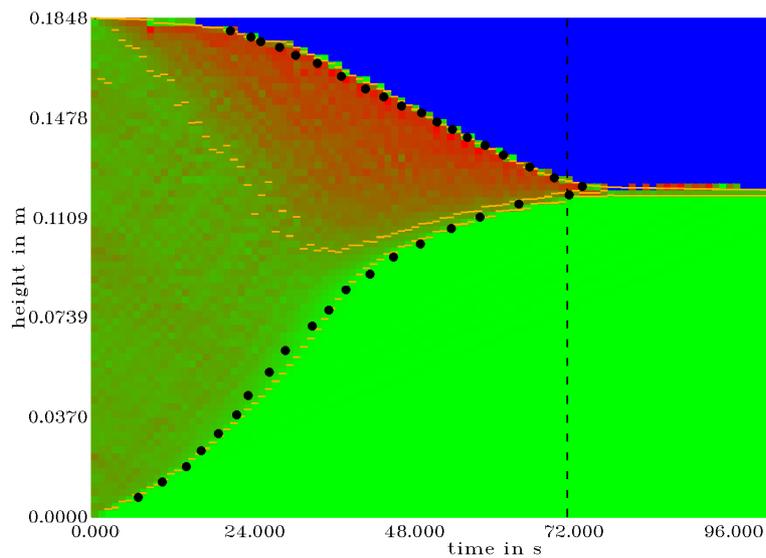
ReDrop: drop-based modeling

definition of system:

- material properties
- process variables
- simulation parameters
- set up arrays and system



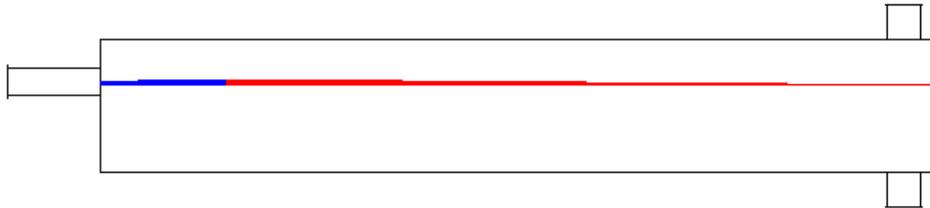
drop-based simulation results



design example

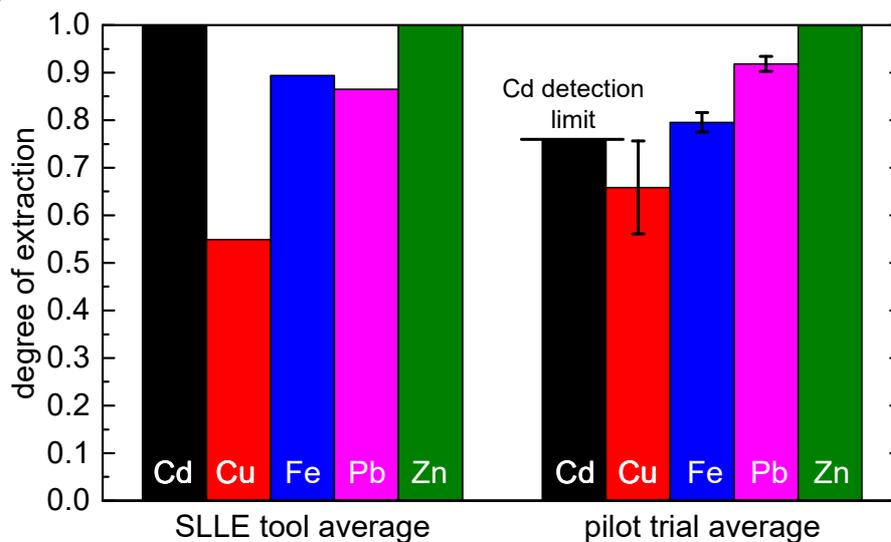
average velocities:

inlet = 35.368 mm/s, in settler = 1.415 mm/s
 top phase = 1.641 mm/s, bottom phase = 1.328 mm/s
 ave. interfacial load with disp. phase = 0.062 mm/s



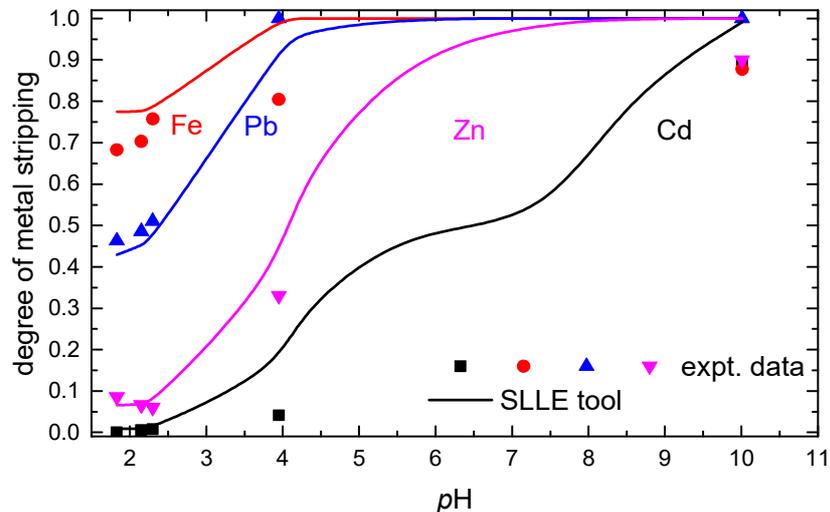
settler length = 1.545 m, settler diameter = 0.250 m
 in-flow length = 0.233 m, coalescence length = 1.311 m

reactive extraction of metals



German sludge
 solvent: Alamine 336,
 TBP & Exxal 10,
 Ketrul D80
 o/a phase ratio: 1.5 to 3

solvent stripping with Ammonia solution

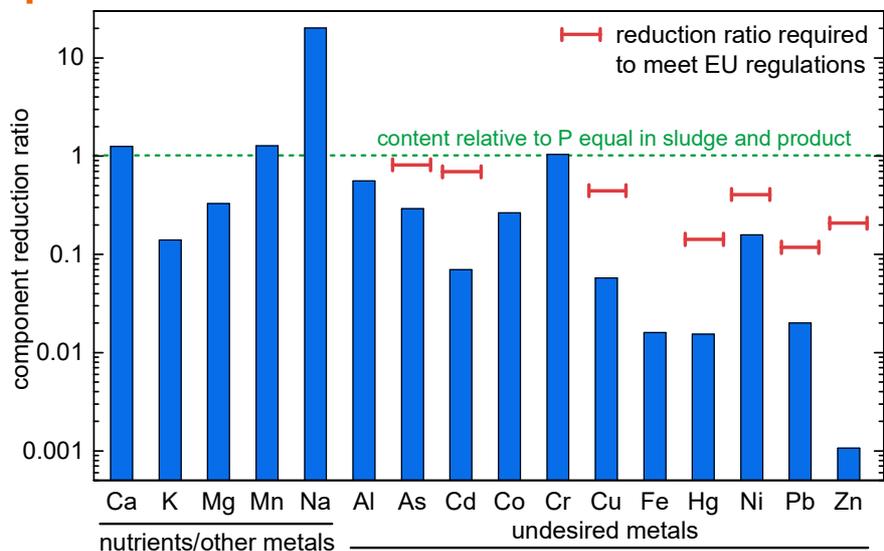


challenges and opportunities



- precipitation during extractant regeneration → challenge in phase separation
- precipitated and dissolved metals obtained during solvent regeneration can be further separated and valorized

metal depollution



final product after precipitation



dried filter cake



granulation of ground product



© Prayon S.A.

pilot trials summary

	German sludge	Belgian sludge	Scottish sludge
sludge source wwtp	Dortmund Deusen	Oupeye	Sterling
sludge	digested 25% DM	undigested 17 to 20% DM	digested 25% DM
P removal	Fe salt precipitation	Bio P	Bio P
operation site	at ULiège	at wwtp	Bo'ness testing facility
quantity treated	340 kg	280 kg	250 kg
P recovery	60 to 65%	60 to 65%	48 to 52%
P ₂ O ₅	33 %	31 %	29 %

summary

- pilot trials conducted with different sludges at different sites
- use of SLLE simulation tool for optimization at each site
 - reduction in experimental work
 - optimization of resource consumption
- solvent extraction → Fe, Cd, Cu, Hg, Pb and Zn extracted
- product analysis and granulation by Prayon → sufficient P₂O₅ content and good granulation, low metal content
- cost: 5 to 10 €-cent per cubic meter of wastewater
- good plant availability of P confirmed by pot trials from UGhent

further process details

- <https://www.nweurope.eu/projects/project-search/phos4you-phosphorus-recovery-from-waste-water-for-your-life>
- search for: phosforyou technical report

acknowledgements

- Interreg North-West Europe and Région Wallonne SPW
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- TOTAL Belgium for providing samples of Ketrul D80
- Exxon Belgium for providing samples of Exxal 10
- UGhent and Prayon Belgium for ICP analyses

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