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Recovery of Phosphorus From Dried Sewage Sludge for Fertilizer Formulations

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agenda

- PULSE process and pilot

- solid-liquid-liquid equilibrium tool

- results: pilot trials and simulation

- summary

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PULSE (Phosphorus University of Liege Sludge Extraction)
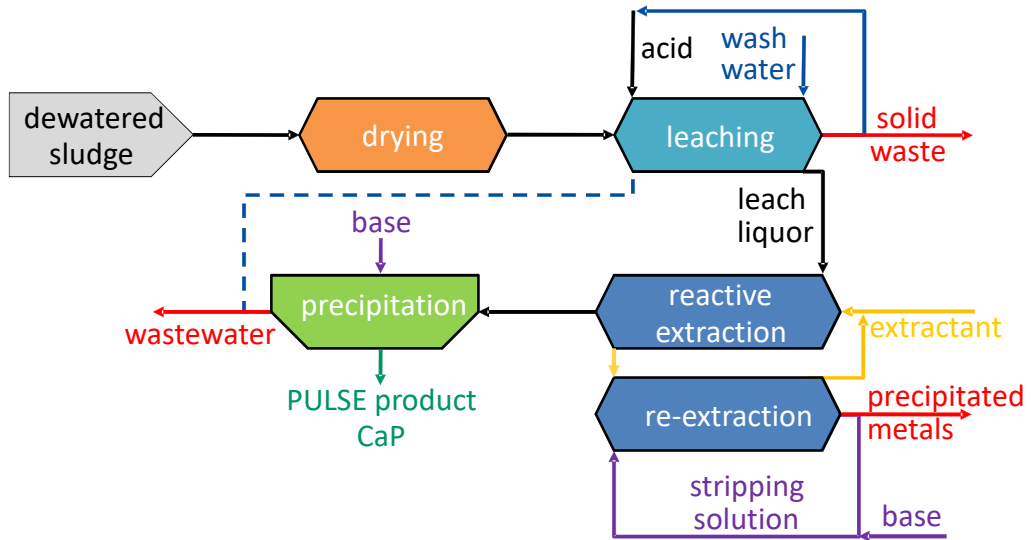
PULSE demonstrator

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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:
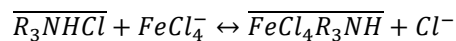 ULiège
  Oupeye WWTP
  Bo'ness, Scotland

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reactions in PULSE process (examples)

- leaching of P and metals from sludge
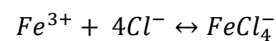
$$FePO_4 + 3HCl \leftrightarrow Fe^{3+} + H_3PO_4 + 3Cl^-$$

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

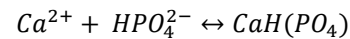- reactive extraction of metals with organic extractant

$$\overline{R_3NHCl} + FeCl_4^- \leftrightarrow \overline{FeCl_4R_3NH} + Cl^-$$

$$K_e = \frac{c_{\overline{FeCl_4R_3NH}}\, c_{Cl^-}}{c_{\overline{R_3NHCl}}\, c_{FeCl_4^-}}$$

- speciation in aqueous solution

$$Fe^{3+} + 4Cl^- \leftrightarrow FeCl_4^-$$

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

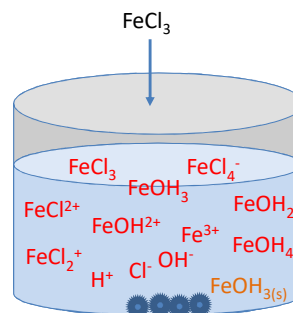- precipitation of CaP

$$Ca^{2+} + HPO_4^{2-} \leftrightarrow CaH(PO_4)$$

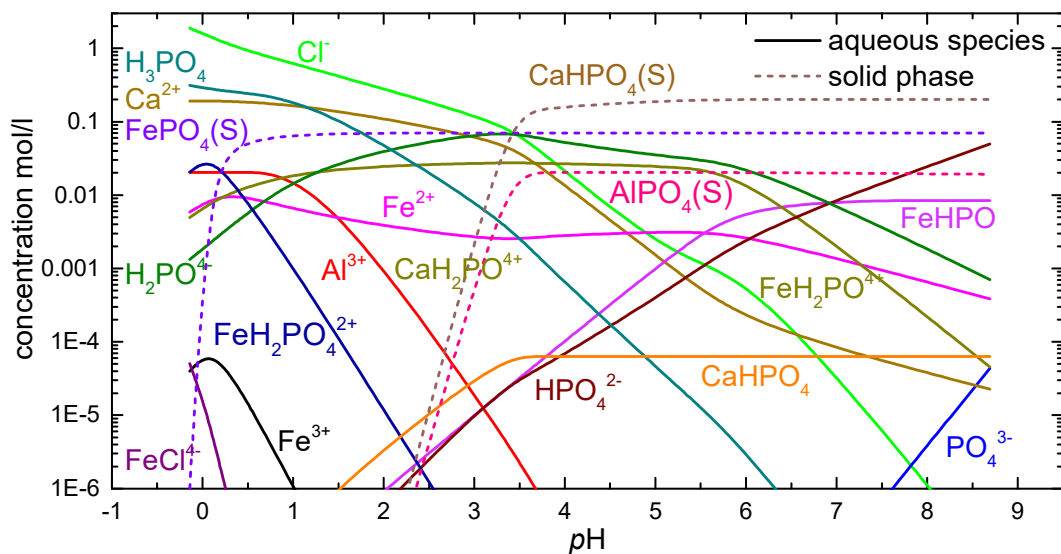$$K_p = \frac{1}{c_{Ca^{2+}}\, c_{HPO_4^{2-}}}$$

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: $IAP = a_A^{\nu_1} a_B^{\nu_2}$
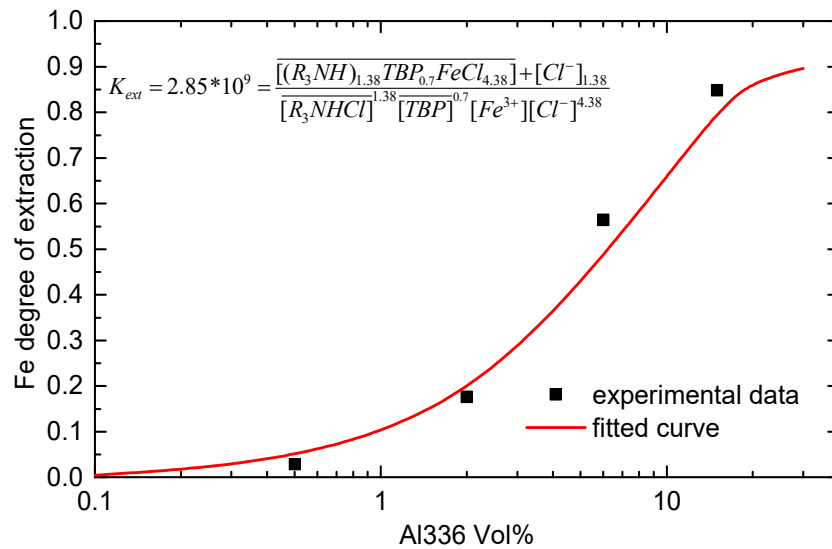
- Saturation Index: $SI = \log IAP - \log K_{sp}$

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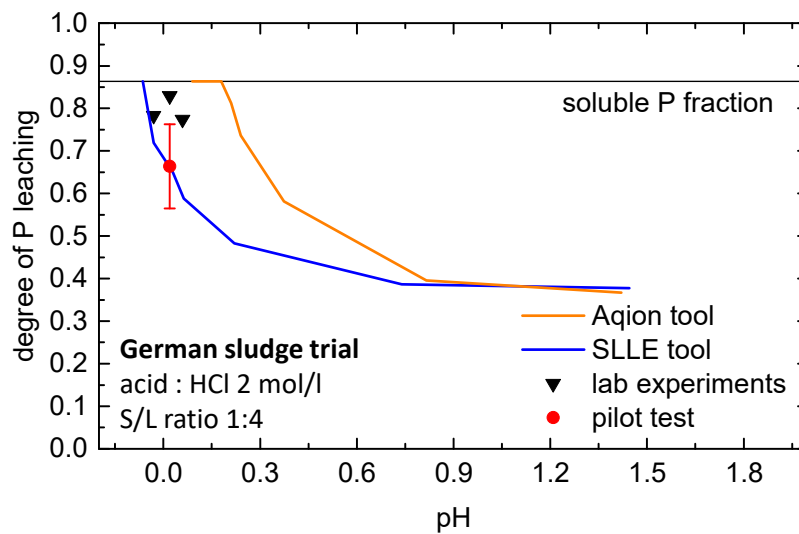c_i = concentration
i = species
j = master species

SLLE tool simulation

parameter fitting with SLLE tool

$$K_{ext} = 2.85 * 10^9 = \frac{\overline{[(R_3NH)_{1.38}TBP_{0.7}FeCl_{4.38}]} + [Cl^-]_{1.38}}{\overline{[R_3NHCl]}^{1.38}\overline{[TBP]}^{0.7}[Fe^{3+}][Cl^-]^{4.38}}$$

Fe degree of extraction vs Al336 Vol%

- ■ experimental data
- — fitted curve

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phosphorus leaching from sludge

degree of P leaching vs pH

soluble P fraction

German sludge trial
acid : HCl 2 mol/l
S/L ratio 1:4

- — Aqion tool
- — SLLE tool
- ▼ lab experiments
- ● pilot test

reactive extraction of metals

degree of extraction

Cd detection limit

Cd Cu Fe Pb Zn
SLLE tool average

Cd Cu Fe Pb Zn
pilot trial average
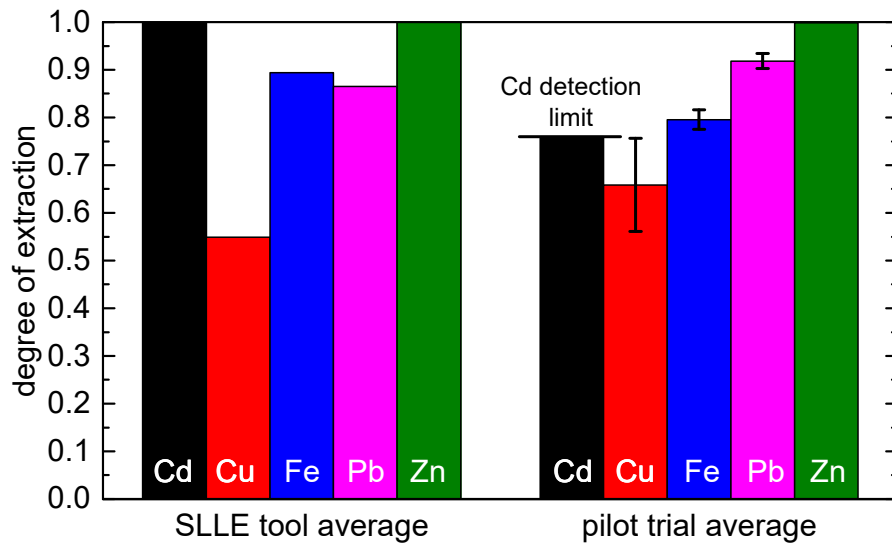
German sludge
solvent: Alamine 336,
          TBP & Exxal 10,
          Ketrul D80
o/a phase ratio: 1.5 to 3

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solvent stripping with Ammonia solution

degree of metal stripping

Fe   Pb   Zn   Cd

■ ● ▲ ▼ expt. data
── SLLE tool

pH

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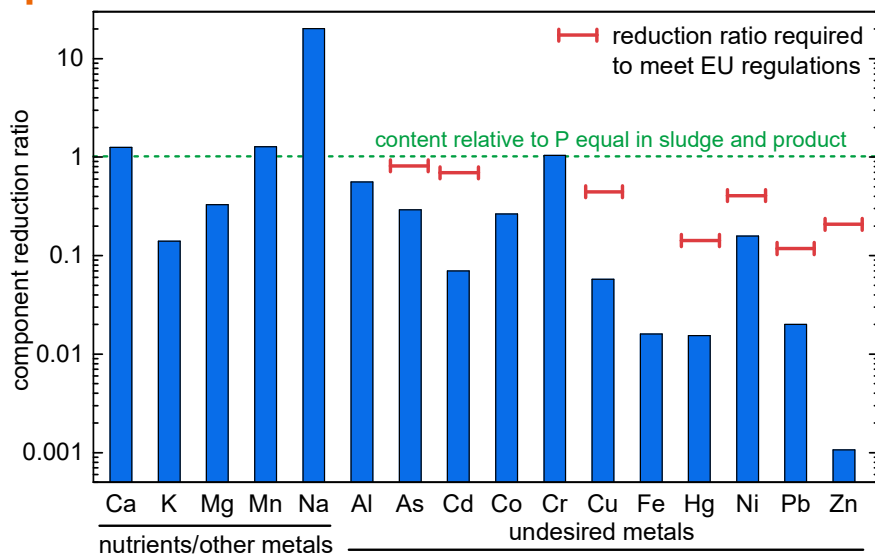Phos4You

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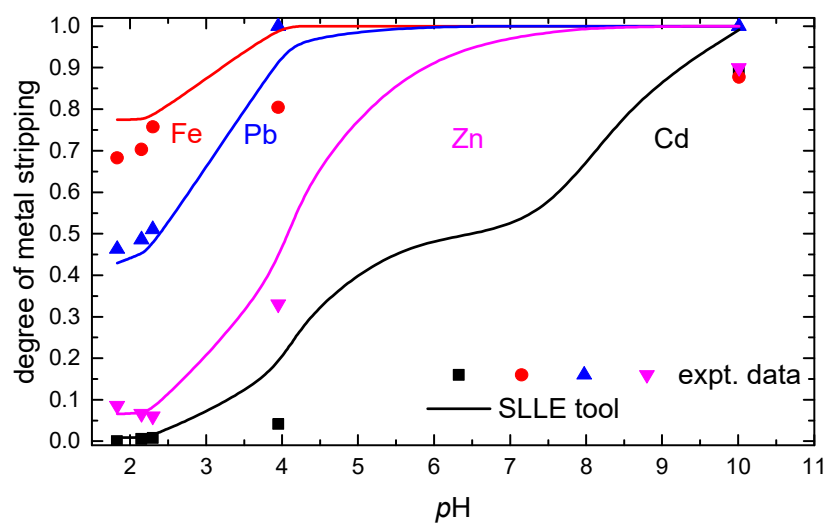
challenges and opportunities

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

metal depollution

reduction ratio required to meet EU regulations

content relative to P equal in sludge and product

component reduction ratio

Ca K Mg Mn Na Al As Cd Co Cr Cu Fe Hg Ni Pb Zn

nutrients/other metals — undesired metals

final product after precipitation

© Prayon S.A.   © Prayon S.A.

dried filter cake   granulation of ground product

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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 P_2O_5 content and good granulation, low metal content
- good plant availability of P confirmed by pot trials from UGhent

acknowledgements

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