

International Solvent Extraction Conference (ISEC 2022)  
Göteborg, Sweden, Sept. 26 to 30, 2022

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## Phosphorous Recovery From Sewage Sludge Utilizing Reactive Extraction

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### agenda

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



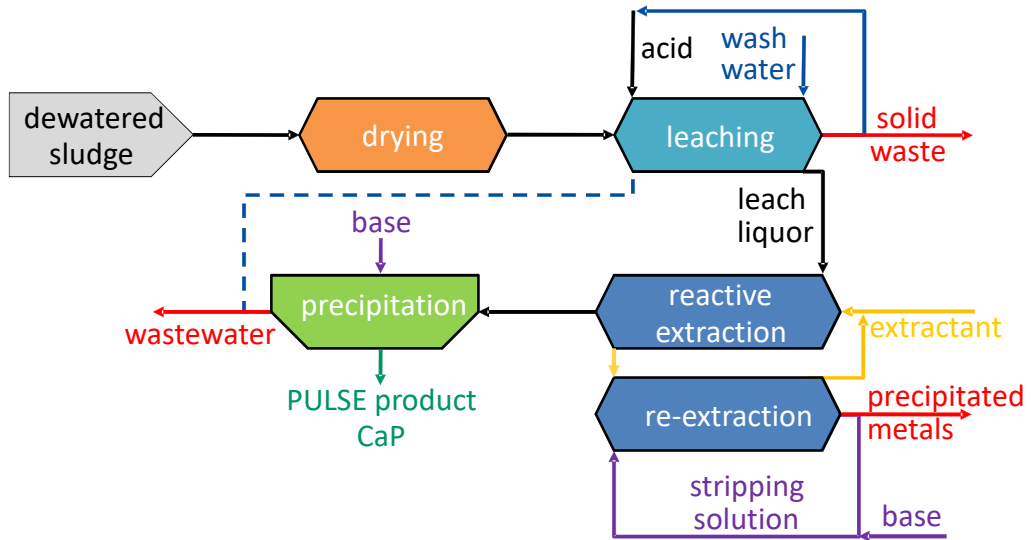
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## PULSE (Phosphorus University of Liege Sludge Extraction)



## PULSE demonstrator



## 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



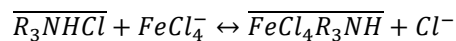
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## reactions in PULSE process (examples)

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

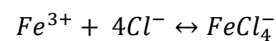
$$K_l = \frac{c_{Fe^{3+}} c_{H_3PO_4} c_{Cl^-}^3}{c_{FePO_4} c_{HCl}^3}$$

- reactive extraction of metals with organic extractant



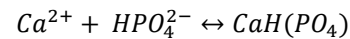
$$K_e = \frac{c_{\overline{FeCl_4R_3NH}} c_{Cl^-}}{c_{\overline{R_3NHCl}} c_{FeCl_4^-}}$$

- speciation in aqueous solution



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

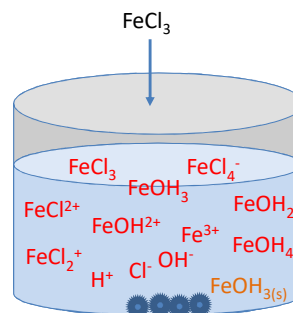
- precipitation of CaP



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

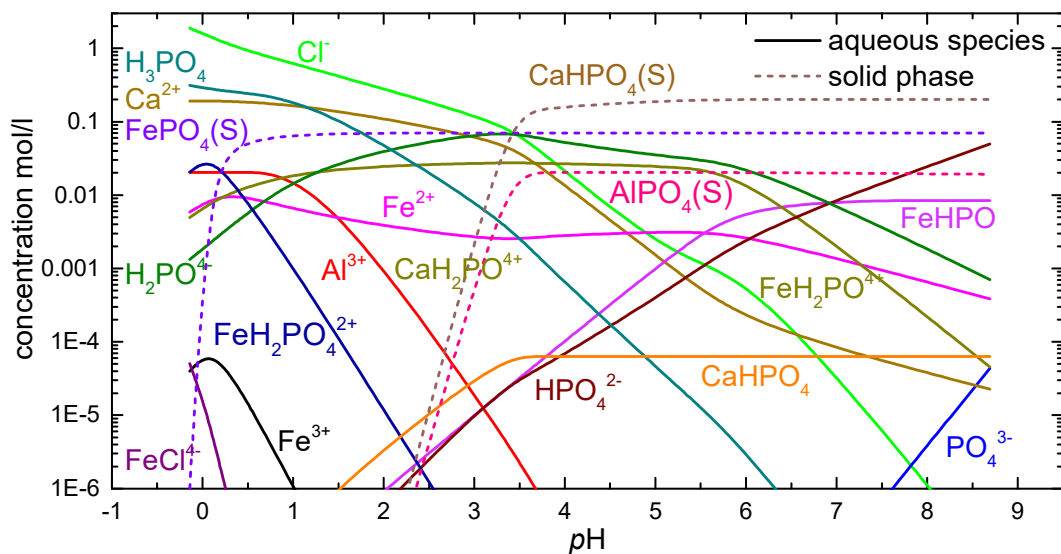
## Solid-Liquid-Liquid Equilibrium tool

- 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:  $IAP = a_A^{v_1} a_B^{v_2}$
- Saturation Index:  $SI = \log IAP - \log K_{sp}$

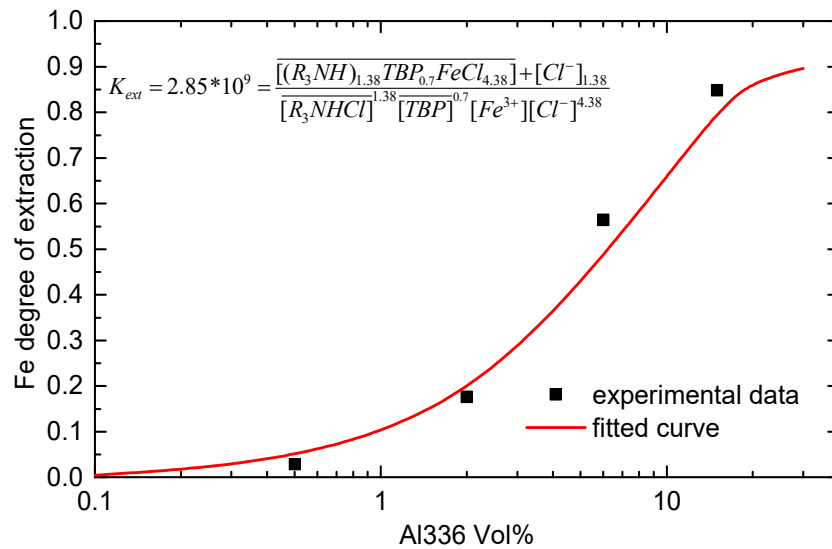


$a_i = \gamma_i c_i$  (activity)  
 $\gamma_i$  = activity coefficient  
 $c_i$  = concentration  
 $i$  = species  
 $j$  = master species

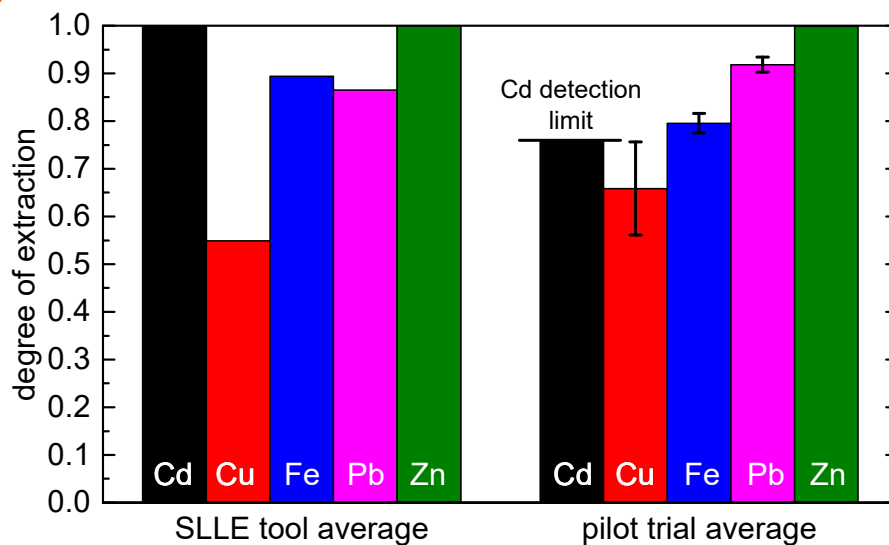
## SLLE tool simulation



## parameter fitting with SLLE tool

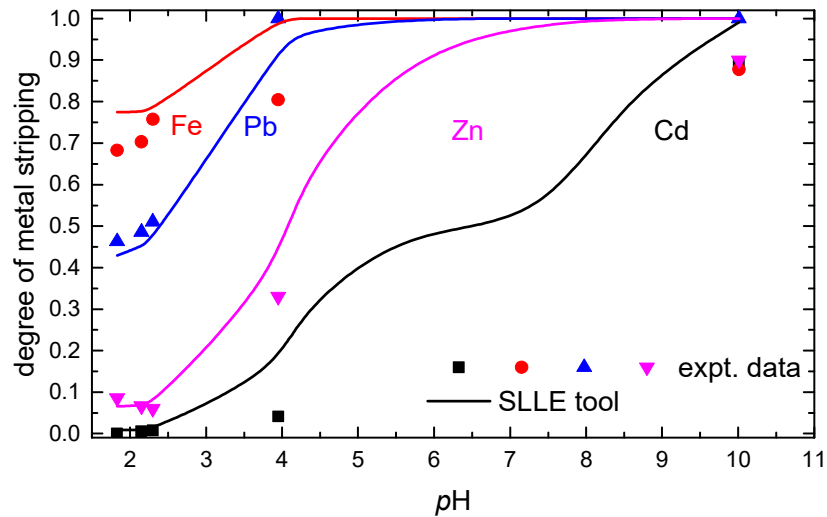


## reactive extraction of metals



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

## solvent stripping with Ammonia solution

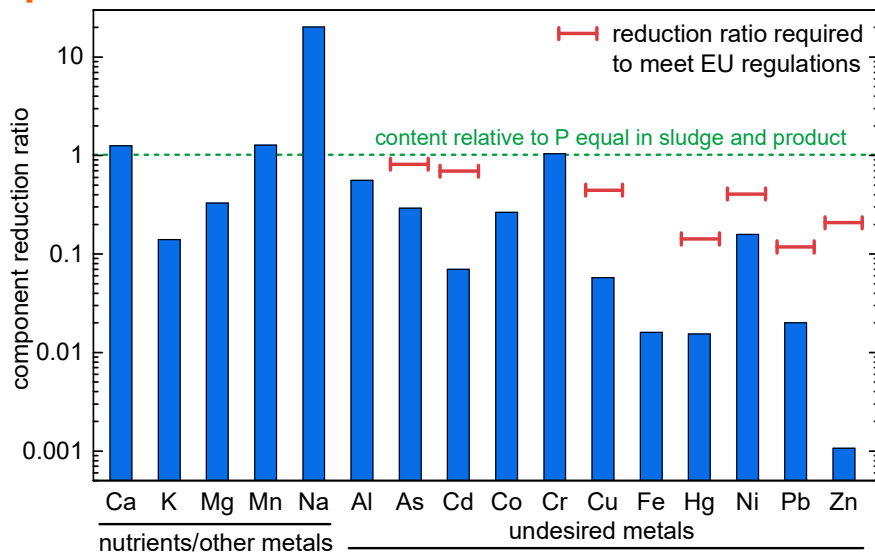


## challenges and opportunities



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

## metal depollution



## 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

- Interreg North-West Europe and Région Wallonne SPW
- BTC Europe GmbH – BASF for providing samples of Alamine336
- 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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