Interreg North-West Europe Phos4You
European Regional Development Fund

Wallonie

LIÈGE université

RESOURCE AND MATERIALS EFFICIENCY

We deliver Phosphorus made in Europe

Recovery of Phosphorus from Sewage Sludge and Subsequent Purification Using Reactive Extraction

17th SFGP Congress, 15-17.10.19, Nantes, France

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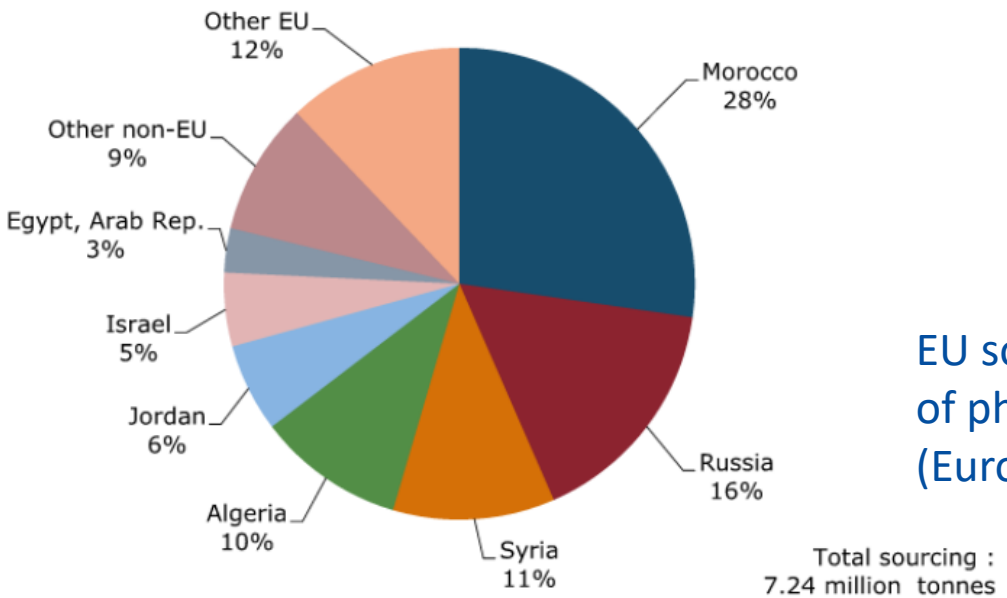
agenda

- introduction
- PULSE process concept
- process development
  - SLE modelling
  - cascaded option tree
- experimental results
- demonstrator
- summary

introduction

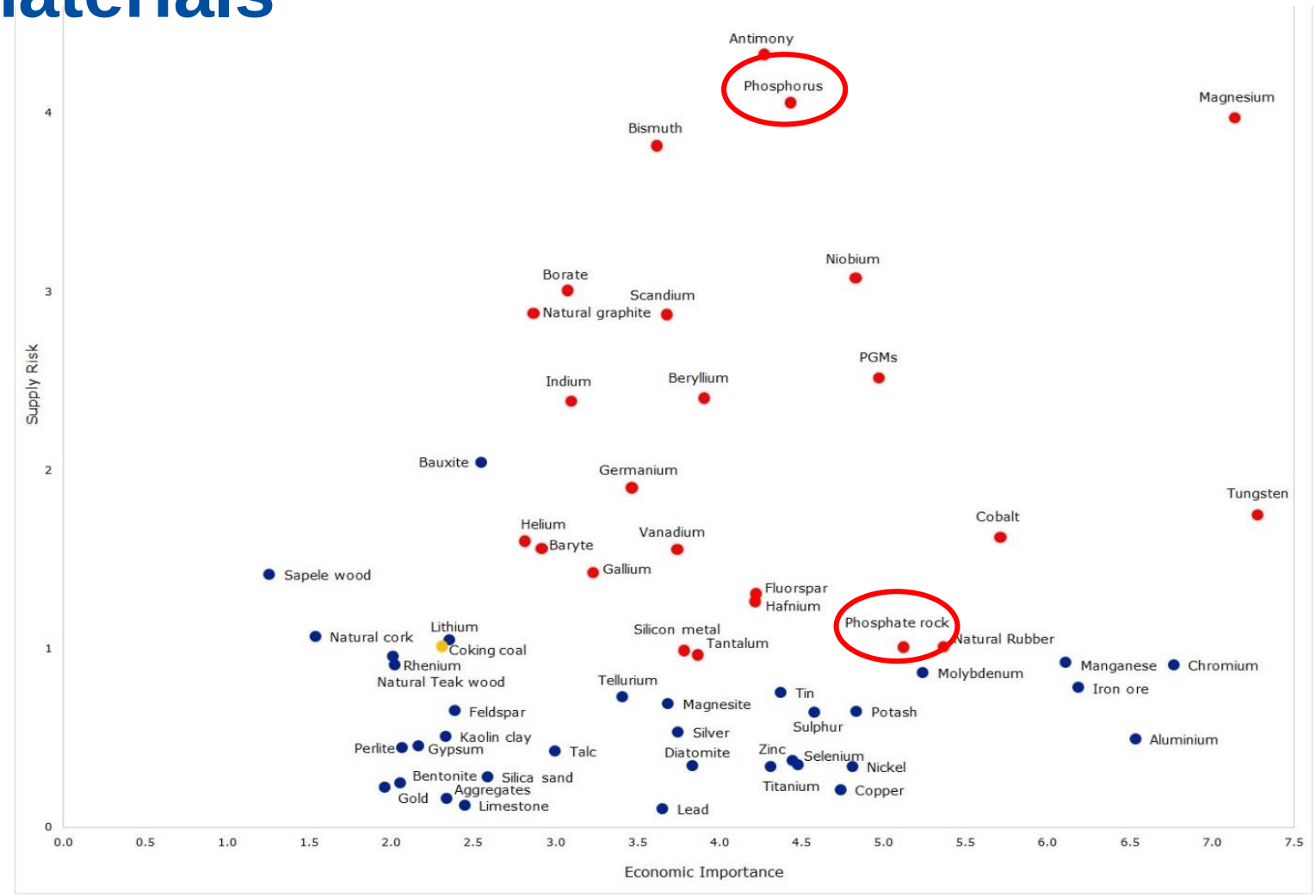
- essential element for all forms of life & finite resource
- more than 80% P produced used in agriculture

rock phosphate



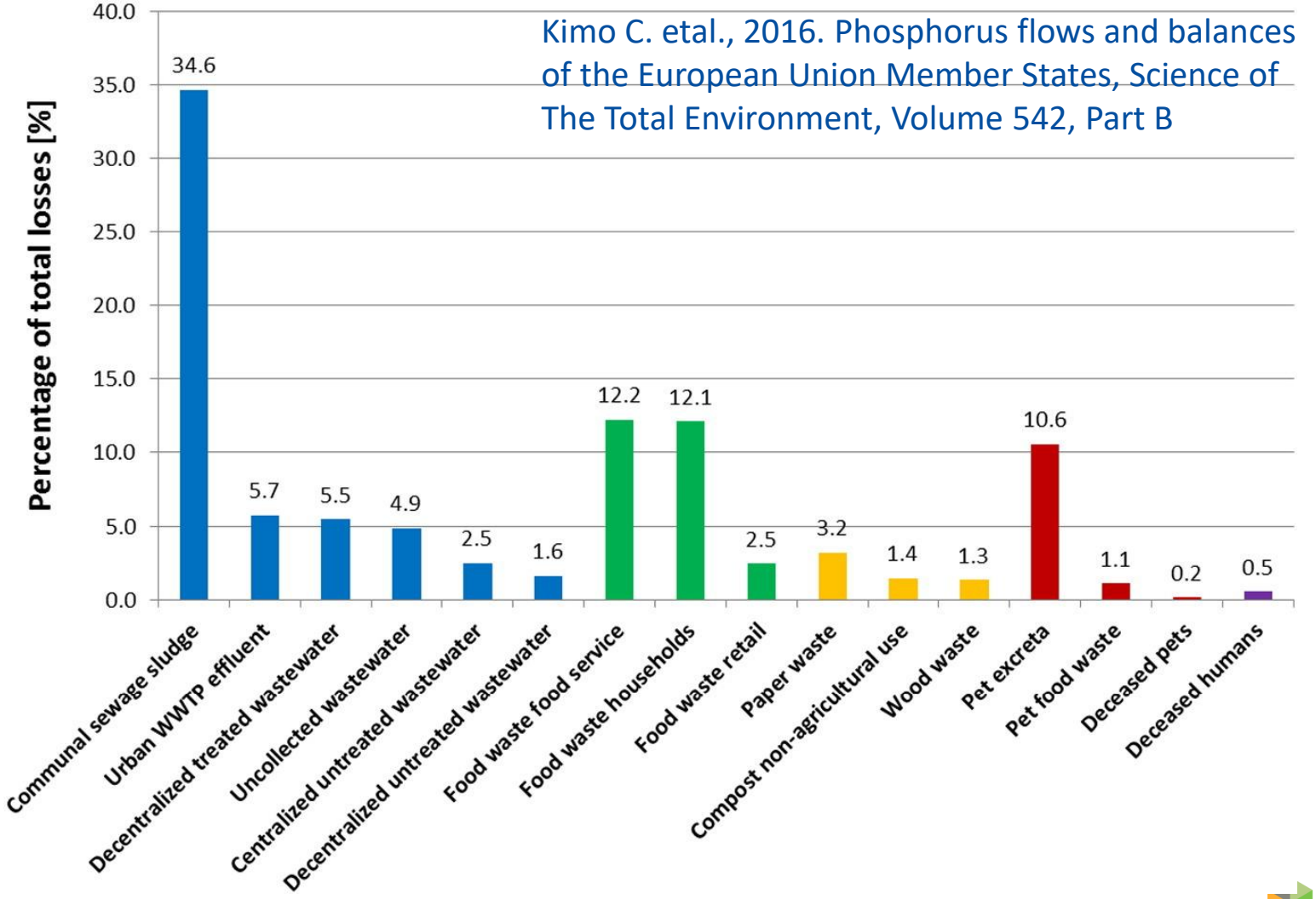
EU sourcing (domestic production + imports) of phosphate rock, average 2010-2014 (Eurostat COMEXT database, 2016)

introduction: EU crictical materials



introduction : fraction of P-loss in waste for EU-27

Kimo C. etal., 2016. Phosphorus flows and balances of the European Union Member States, Science of The Total Environment, Volume 542, Part B

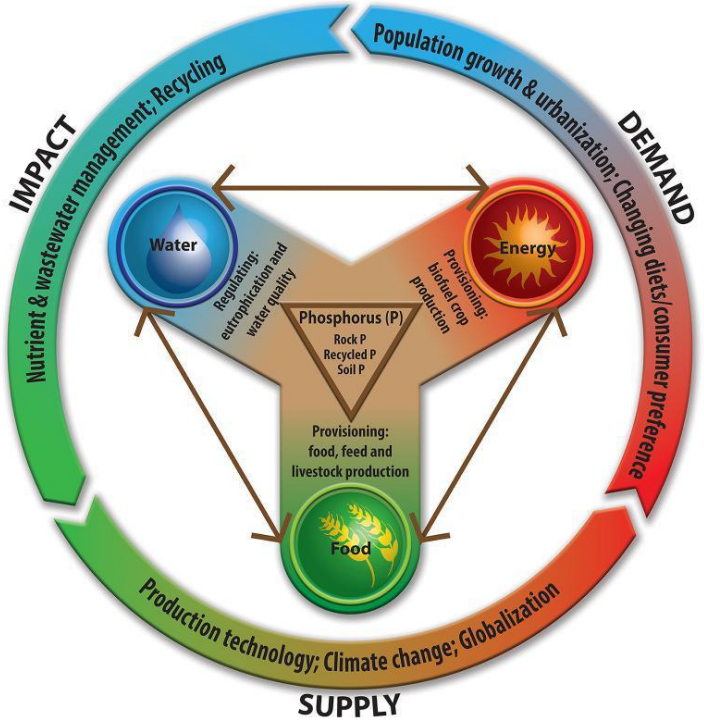


introduction : P recycling

- P-recovery potential from sewage sludge for EU > 25%
- German legislation (2017) – either recovery of P > 50% or reduction of P in sewage sludge to <2% by 2029
- new EU fertilizer regulation: certain types recycled P allowed to be used in fertilizers (published June 2019)
  - first EU product legislation to confer “End of Waste” status

introduction: Phos4You project

- accelerate the exploitation of P recovery in sewage and close the gap between P-recovery and recycling.
- 12 partners from 6 EU member states and Switzerland
- demonstrate 7 P-recovery technologies
- ULiège: demonstrate PULSE process and LCA for all the technologies

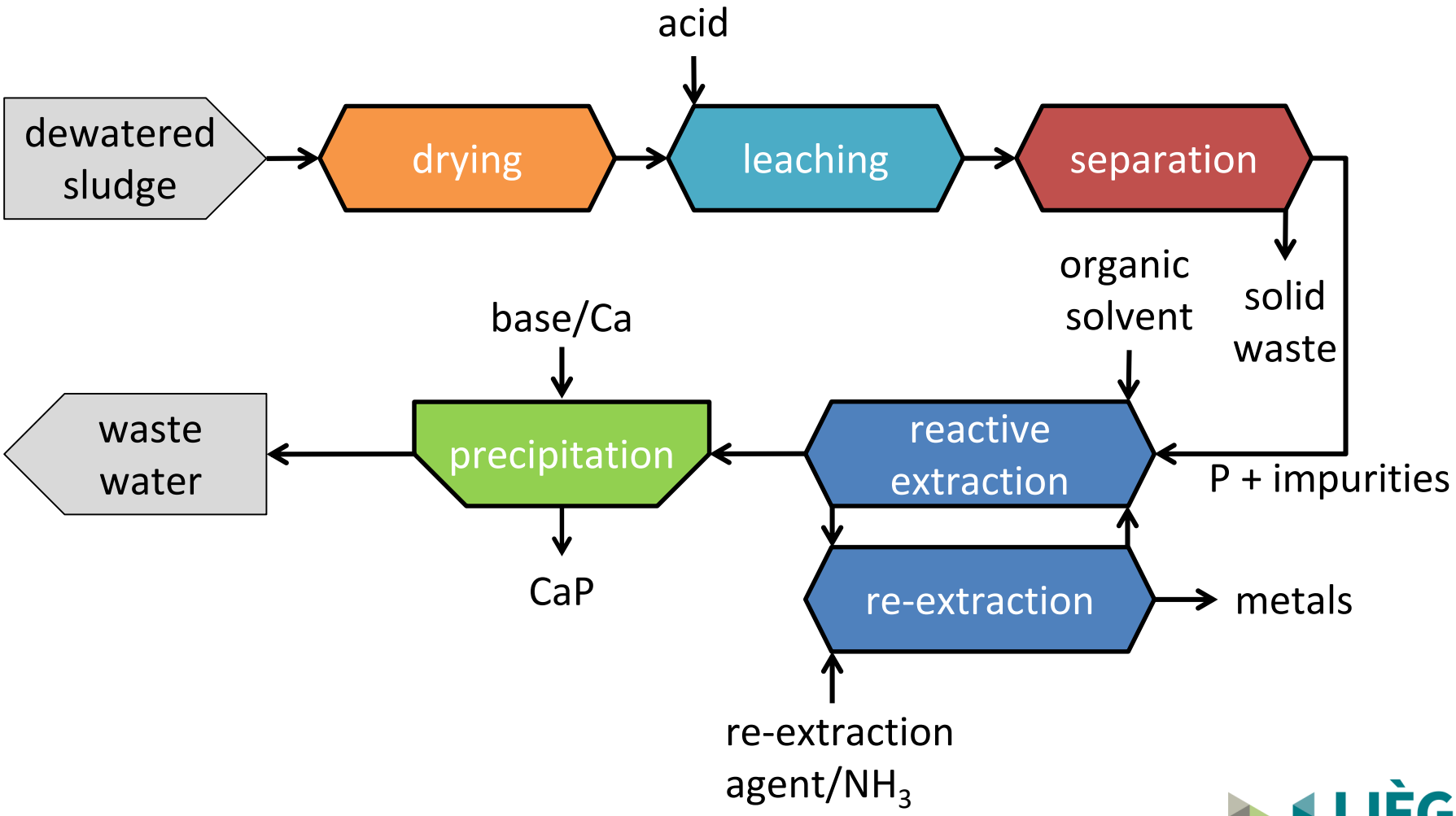


introduction : Phos4You

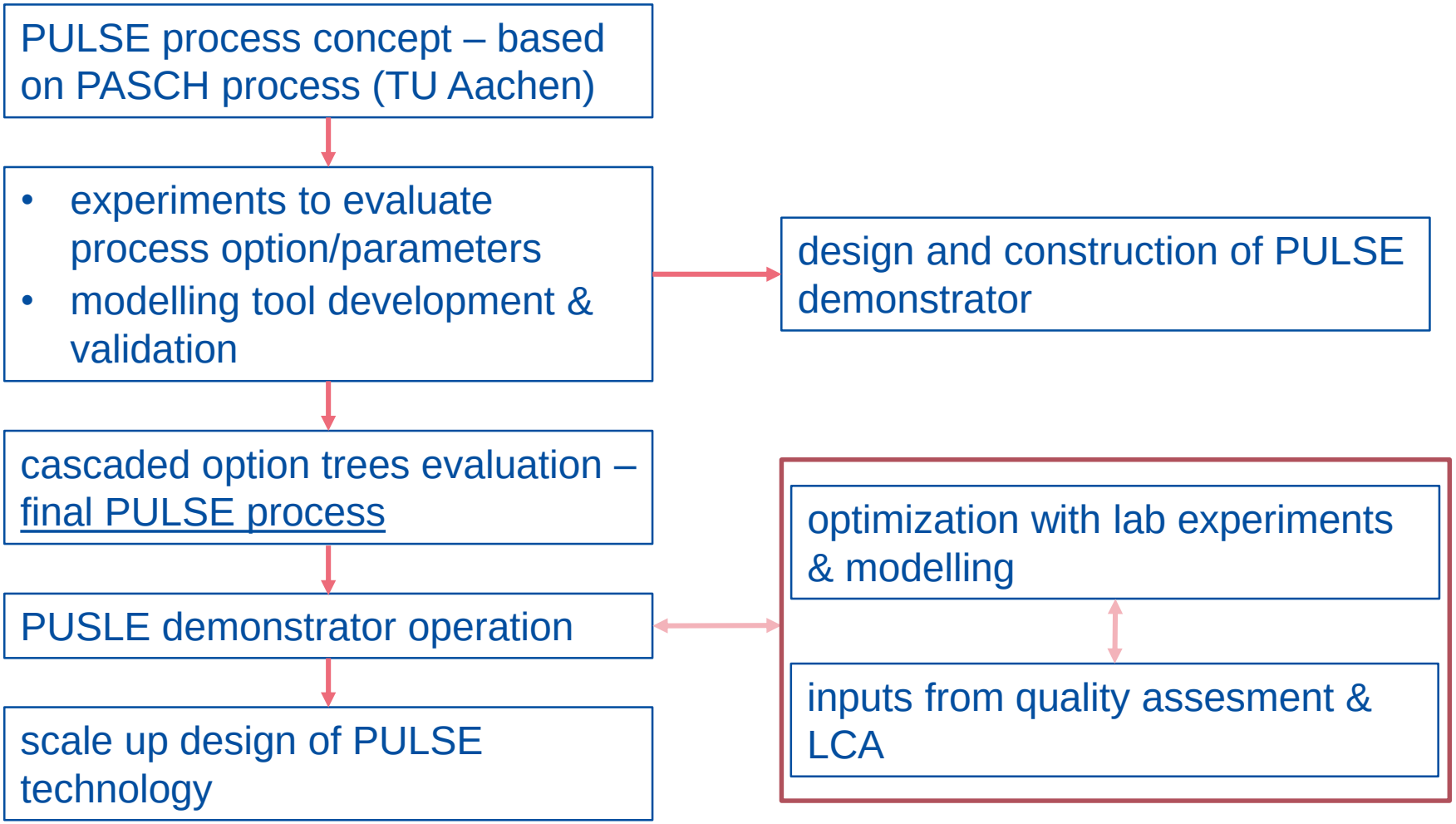
demonstrator	country	source	process
EuPhoRe GmbH	Germany	(digested) sludge	pyrolysis followed by incineration
Lippeverband with REMONDIS Aqua	Germany	sewage sludge ashes	chemical leaching followed by ion-exchange
PULSE process, ULiége	Belgium	dried sludge	chemical leaching, reactive extraction, precipitation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

CIT: Cork Institute of Technology; GCU: Glasgow Caledonian University; ERI: Environmental Research Institute

PULSE (Phosphorus University of Liege Sludge Extraction) process



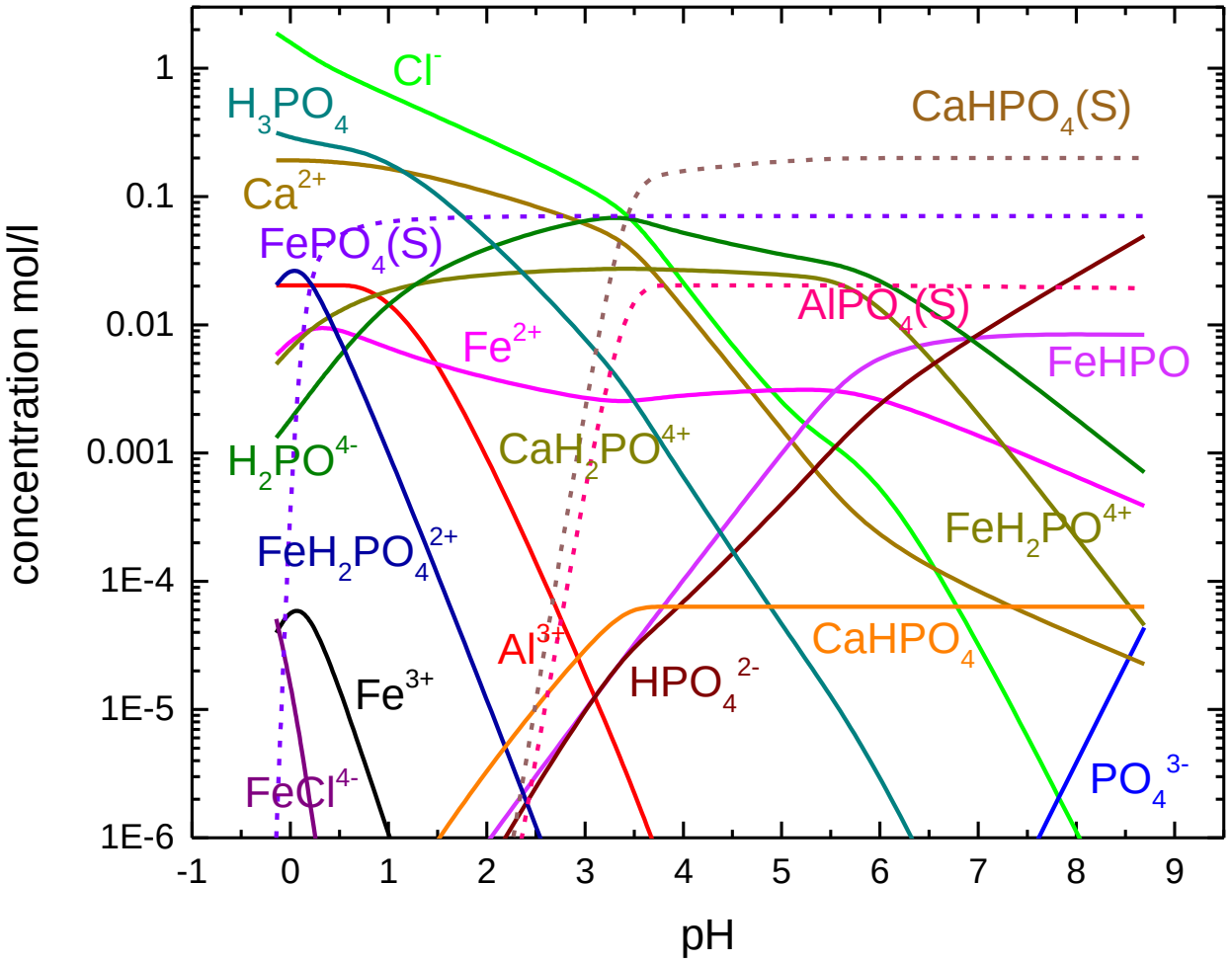
PULSE process development



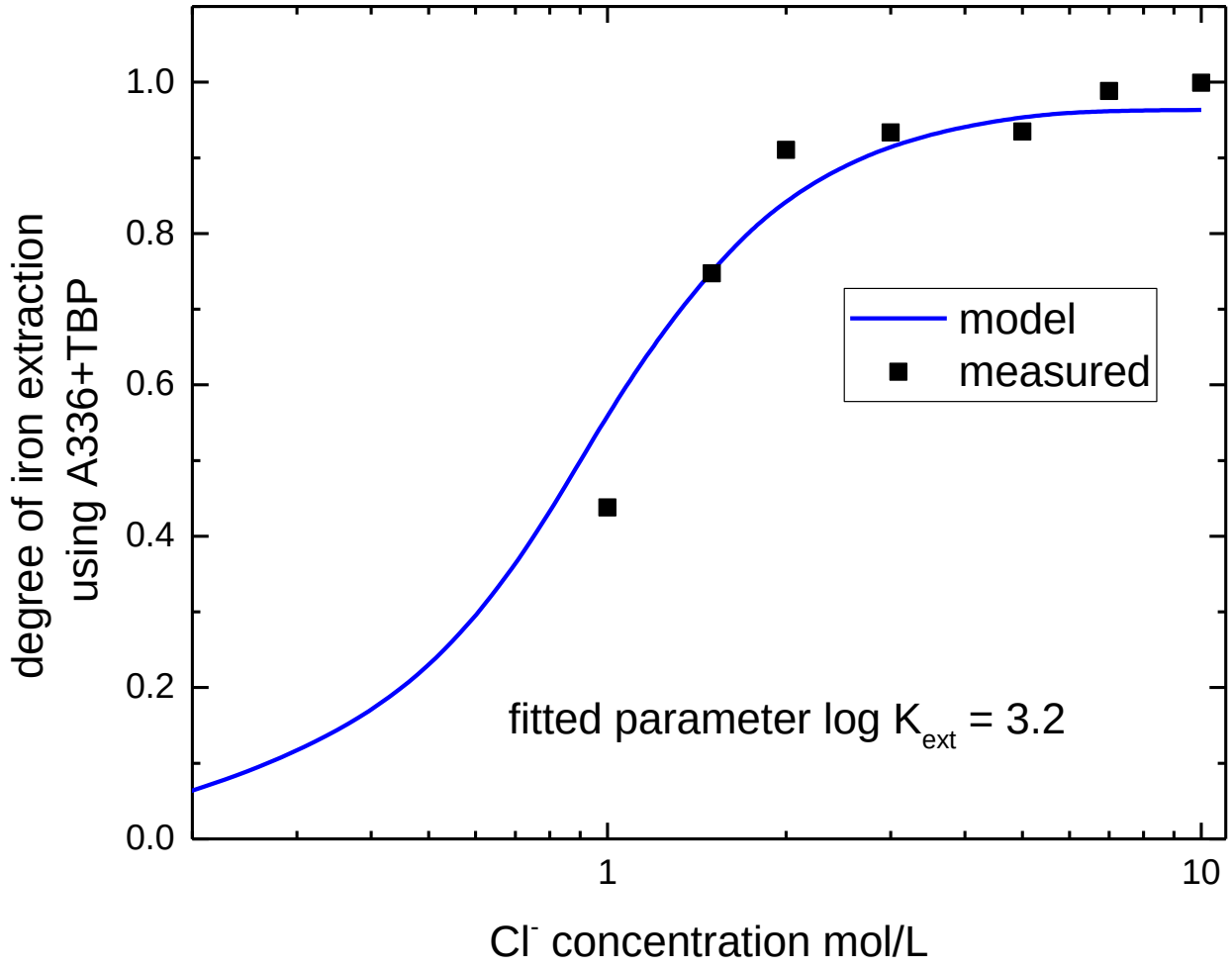
modelling: Solid-Liquid-Liquid Equilibrium

- aqueous phase speciation:
  - mass balance
  - law of mass action
  - charge balance
- solid precipitation:
  - saturation index
- activities instead of concentration to account for ionic interactions
- efficiency of reactive extraction with organic solvents
  - determination of extraction parameters by data fitting

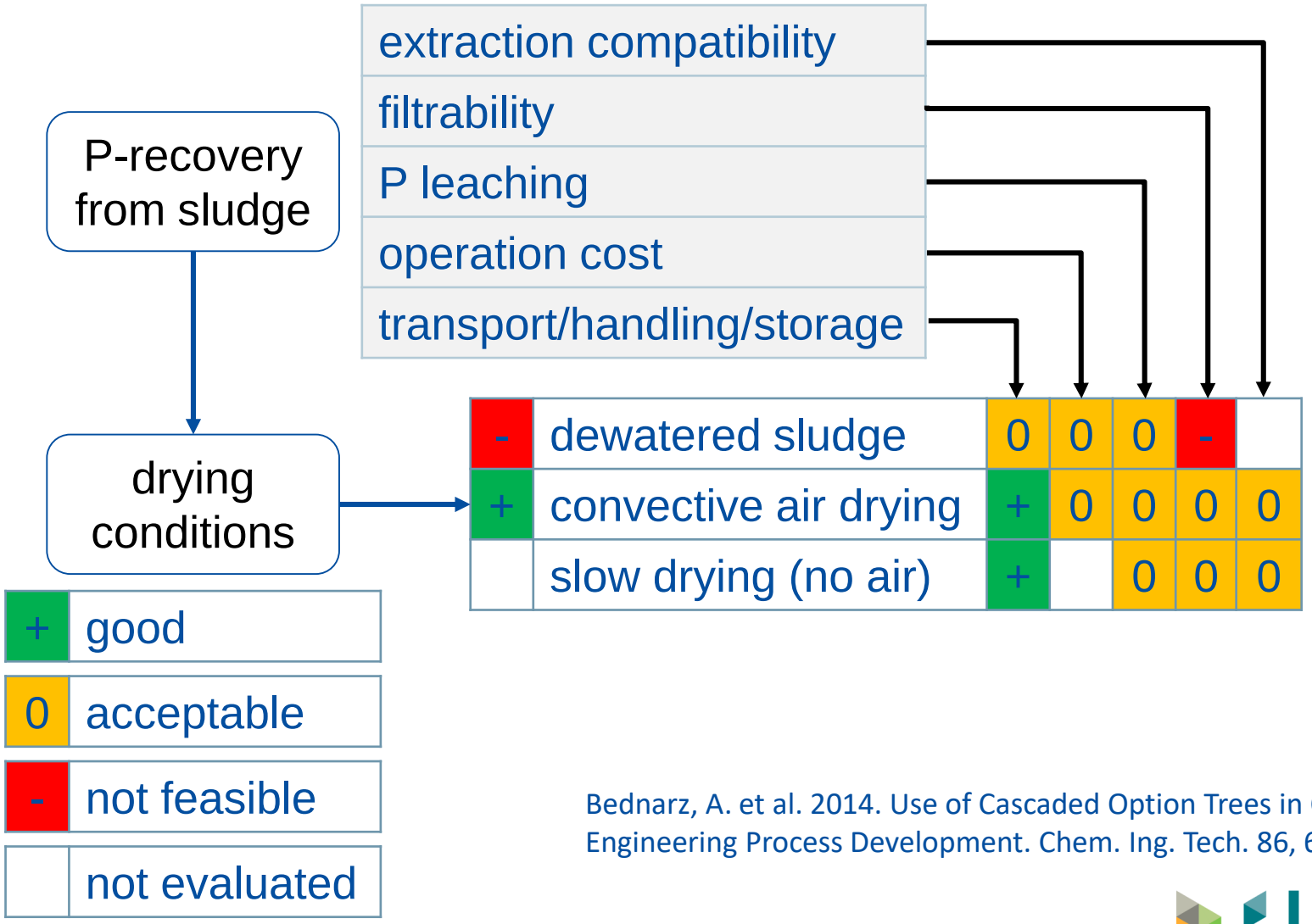
modelling: SLE & speciation



modelling tool: non-linear data fitting



cascaded option tree: drying



Bednarz, A. et al. 2014. Use of Cascaded Option Trees in Chemical-Engineering Process Development. Chem. Ing. Tech. 86, 611-620

sludge drying

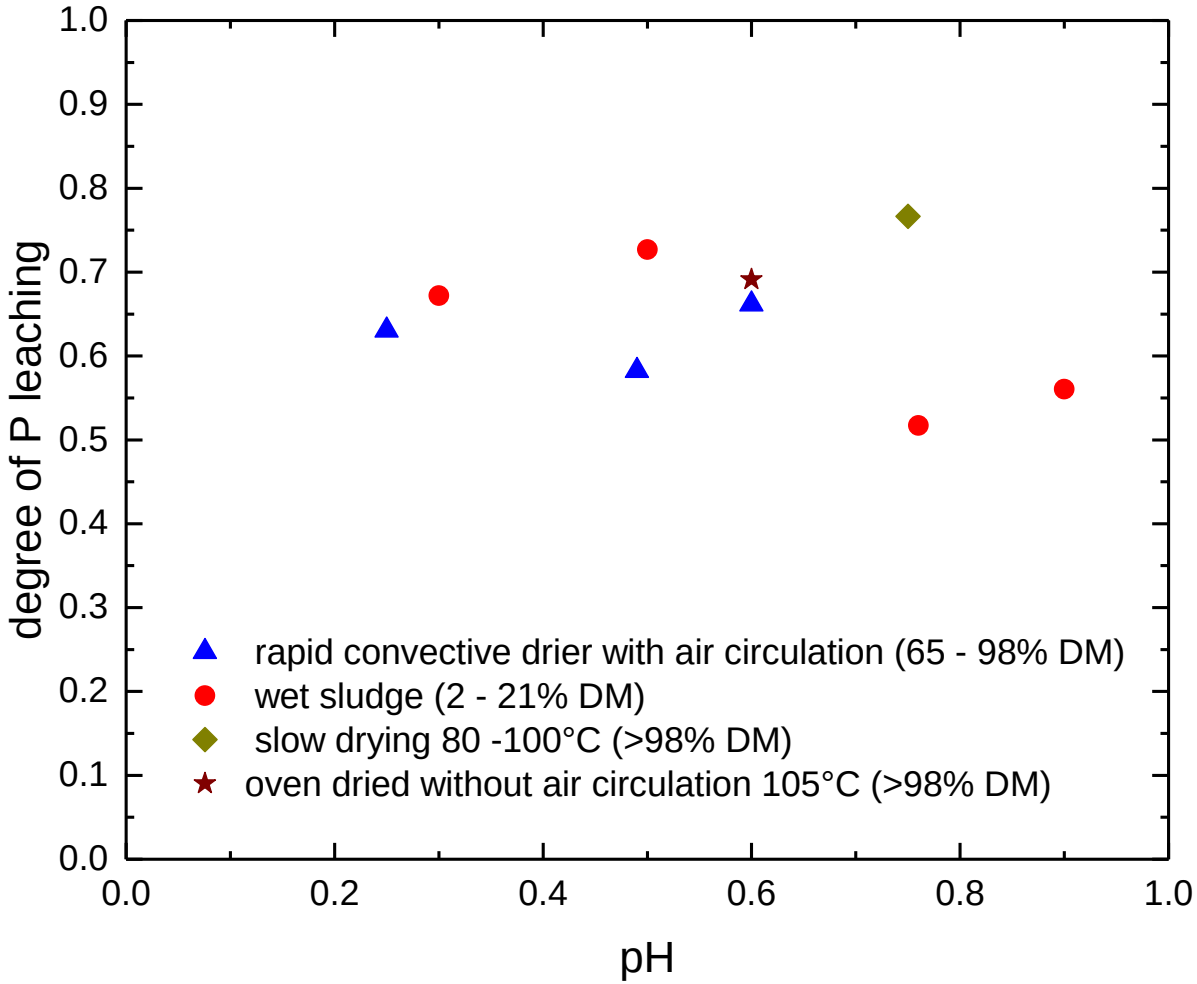
dewatered sludge (20-22% DM)

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

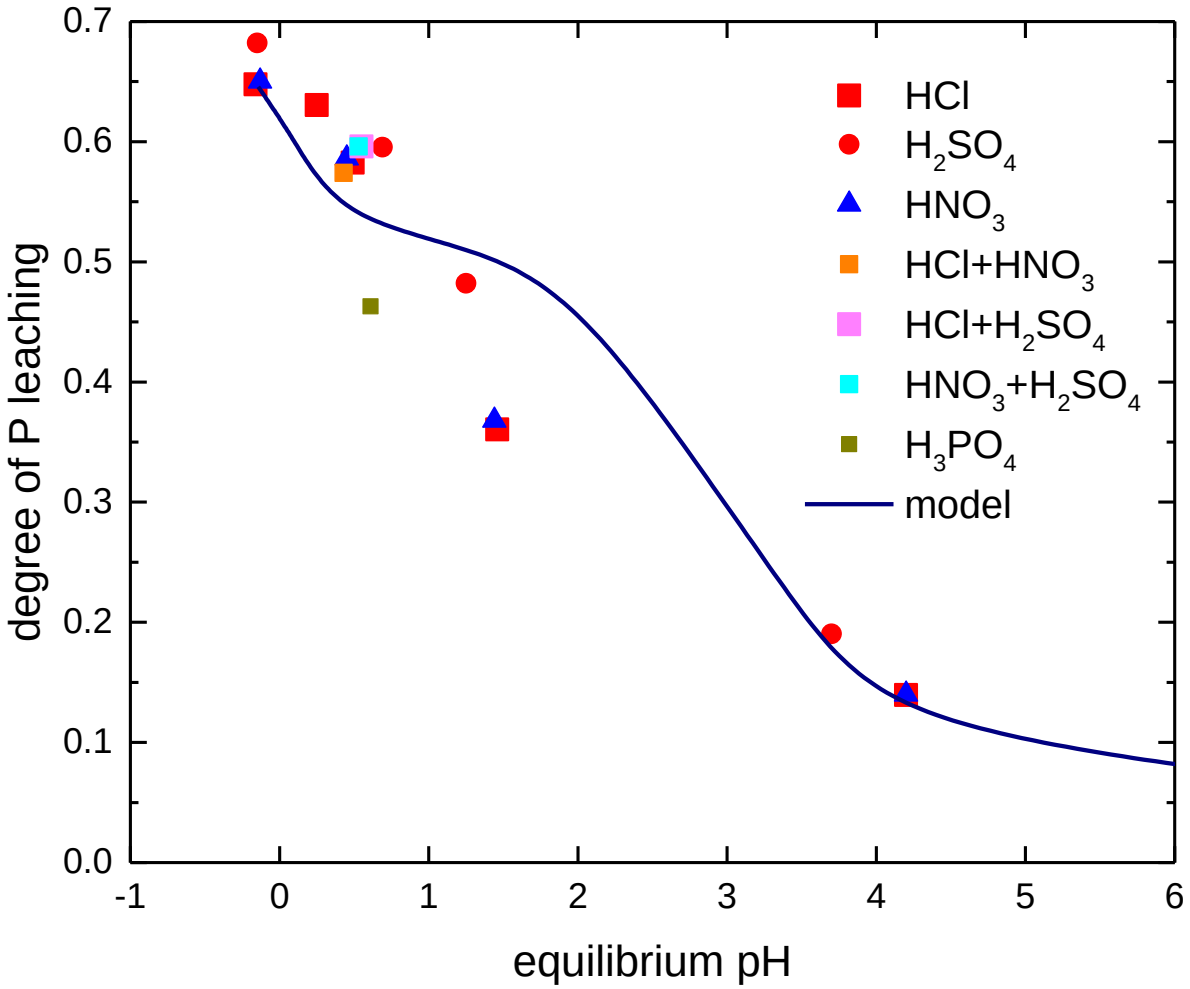
dried sludge (>95% DM)

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

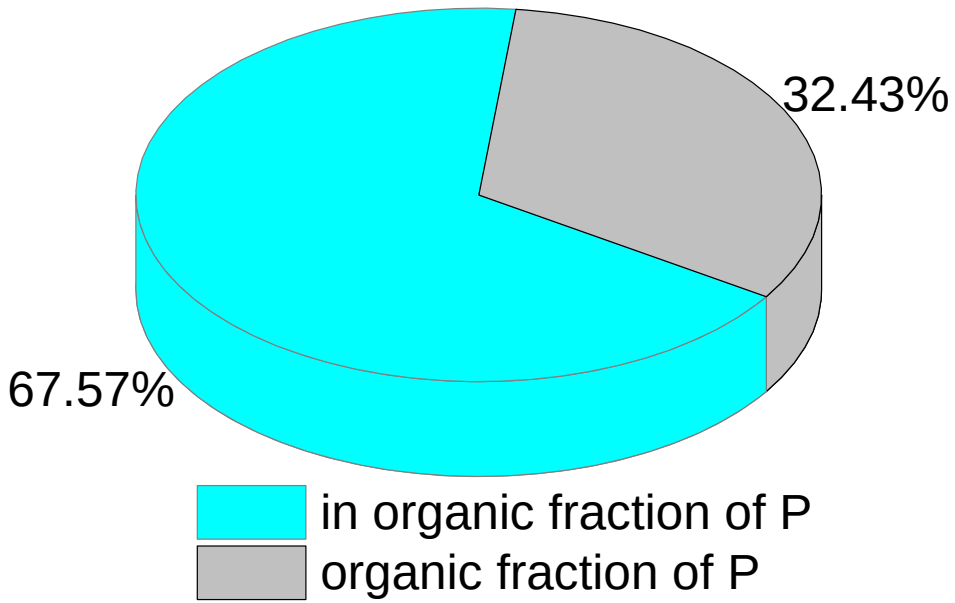
P leaching- wet/dry sludge



P leaching- different acids and pH

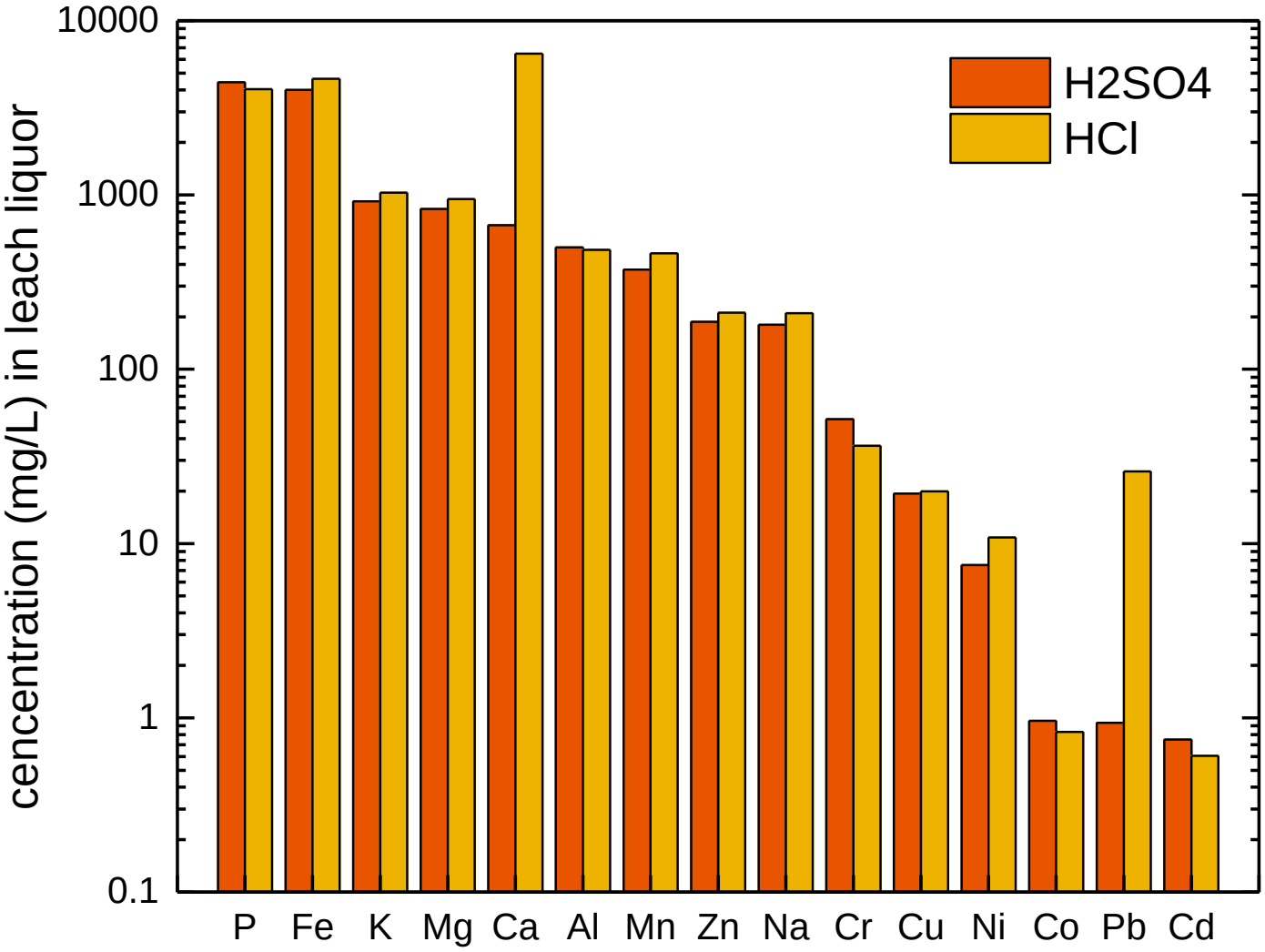


P leaching- organic & inorganic P

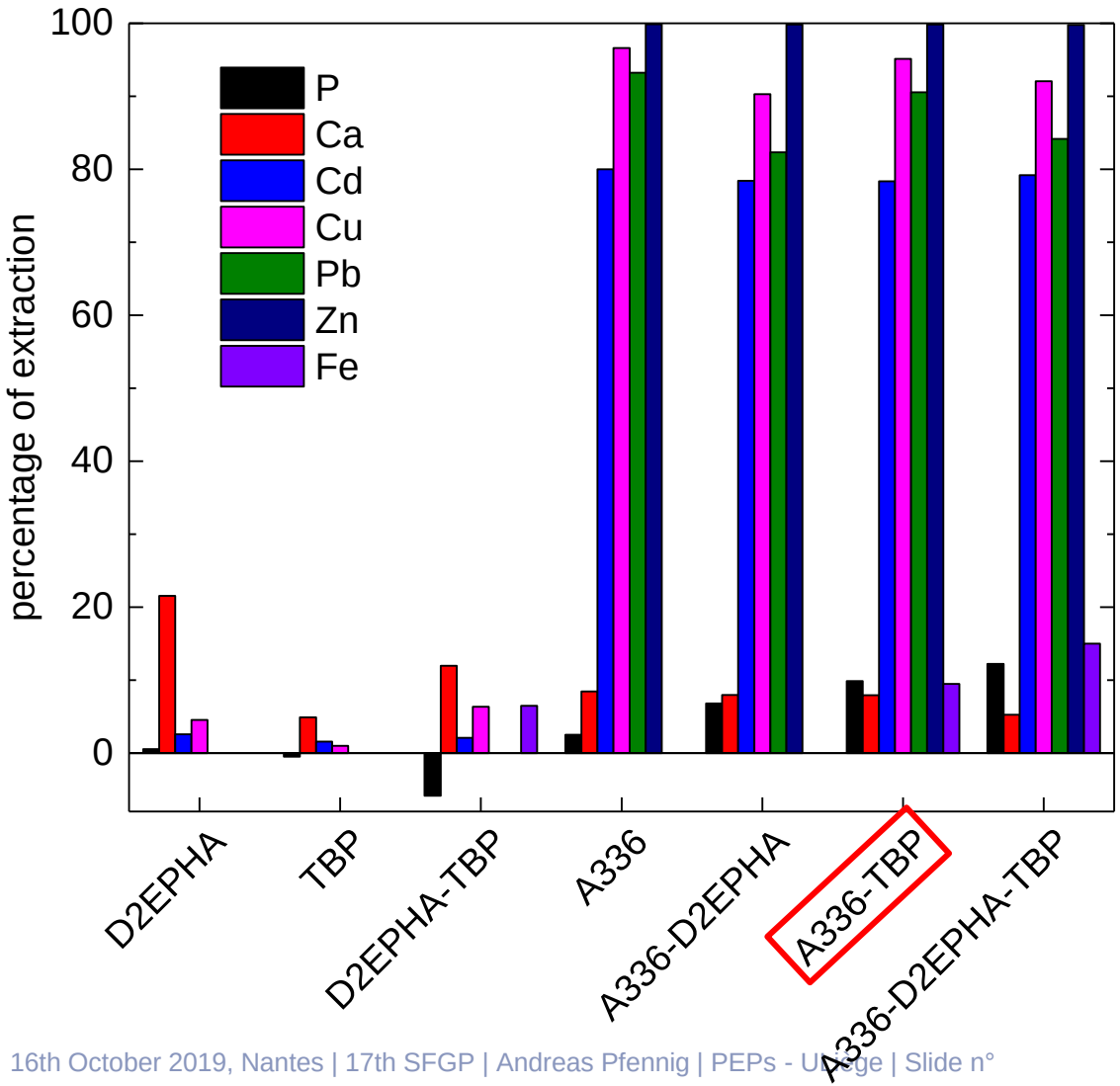


*According to The standards, measurements, and testing (SMT) procedure for phosphorus fractionation in freshwater sediments, developed within the framework of SMT Program of the EC

sludge leach liquor



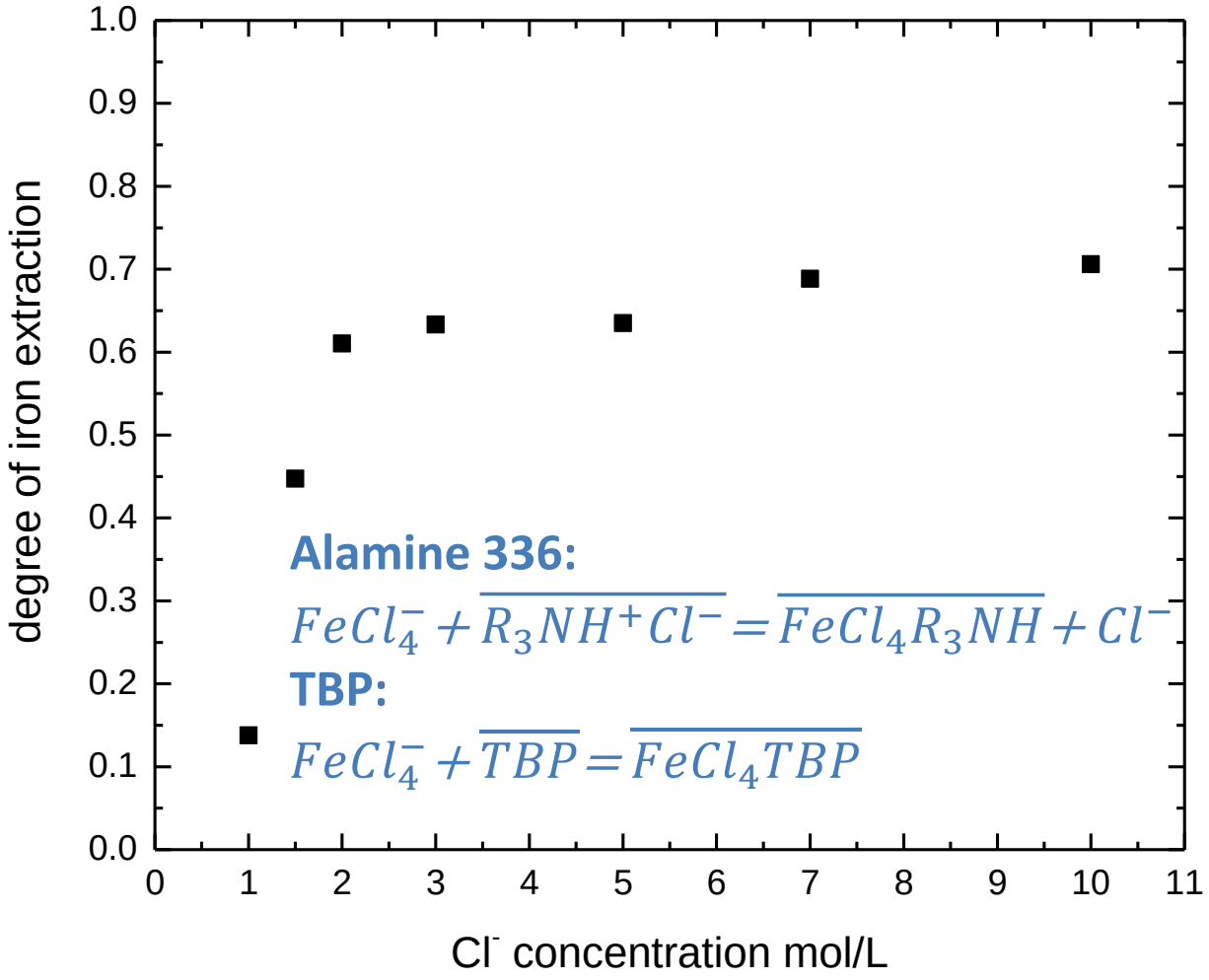
reactive extraction with HCl conc. 1mol/L



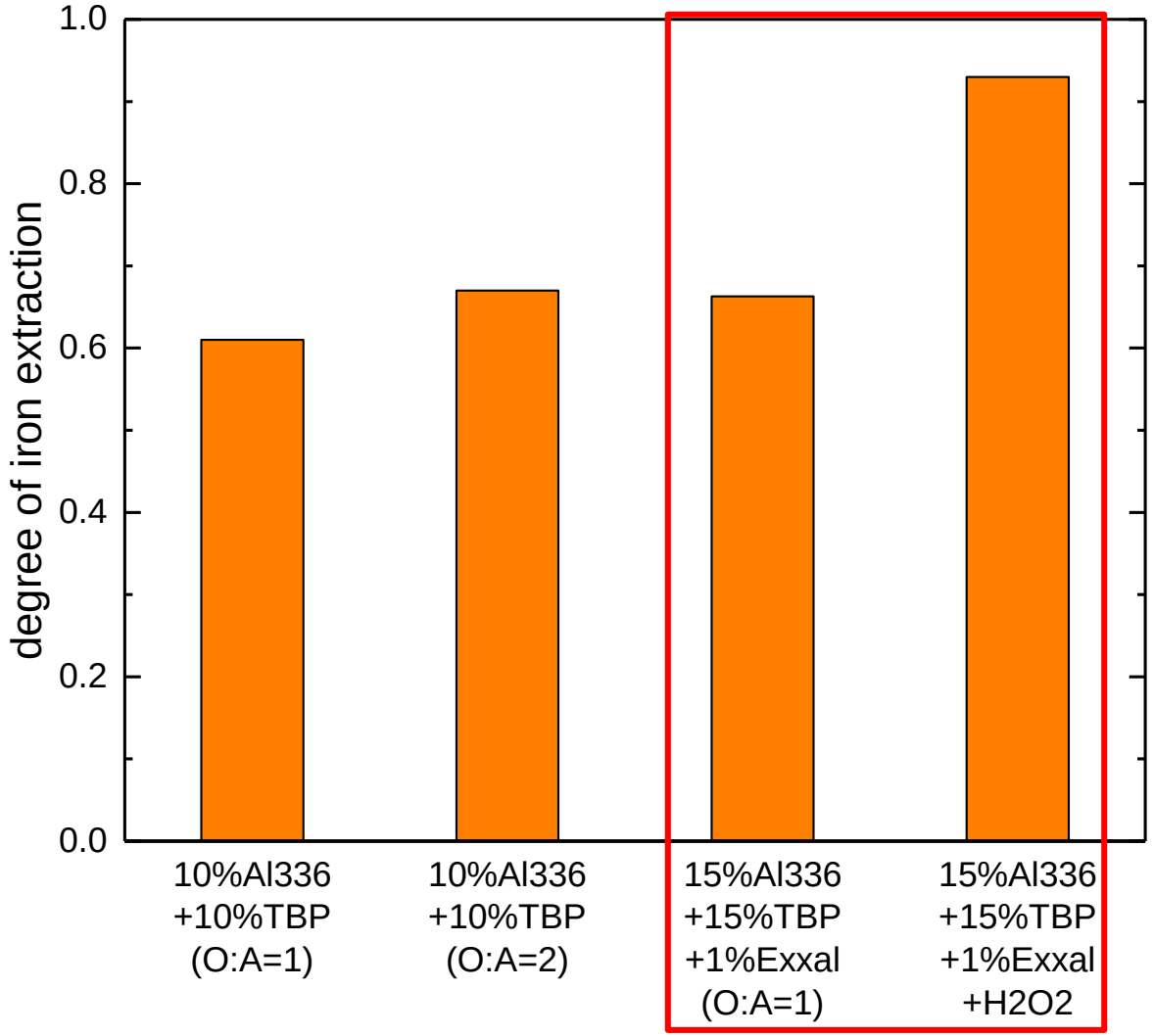
mixing

settling

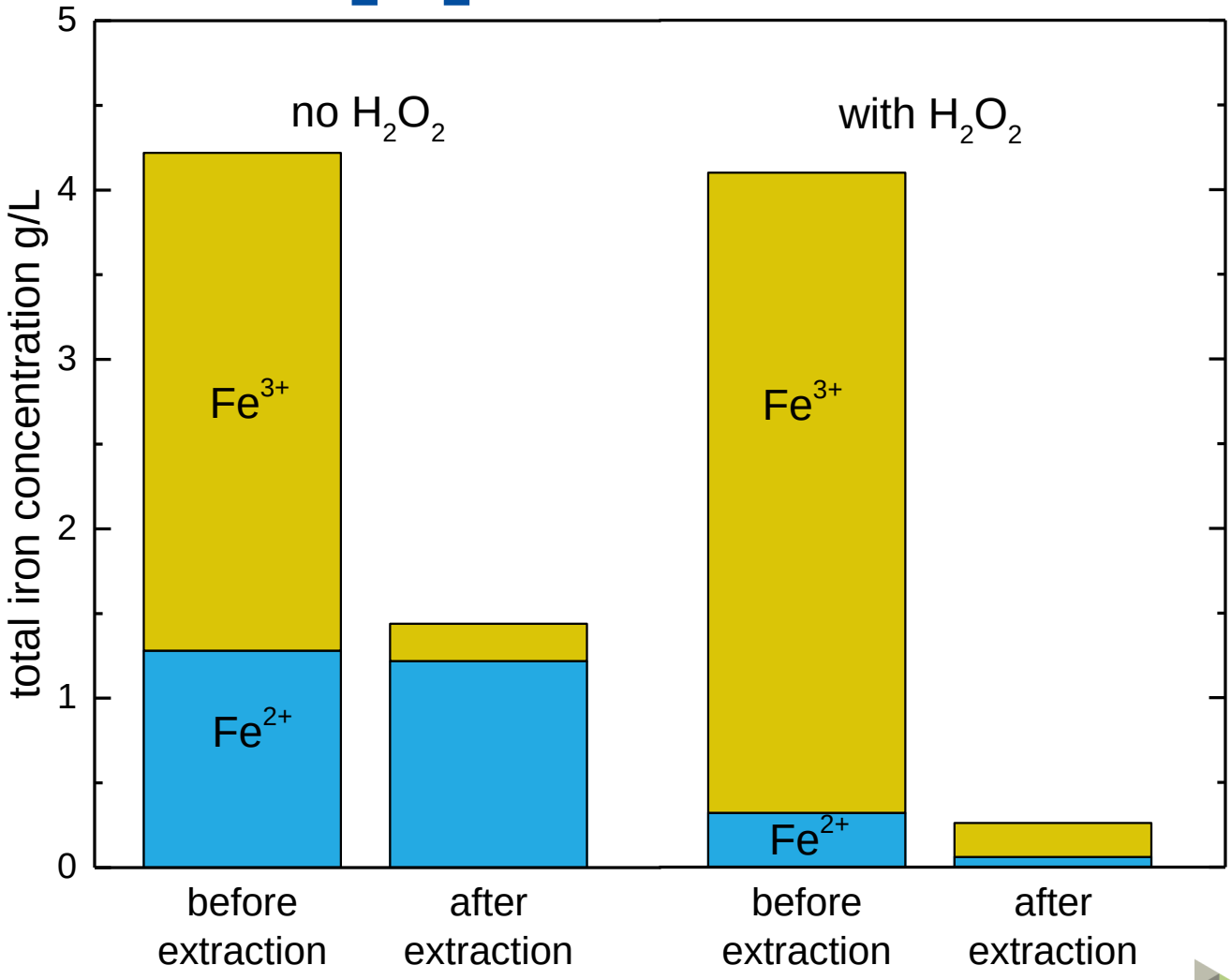
Fe extraction with A336-TBP at varying Cl^- concentrations



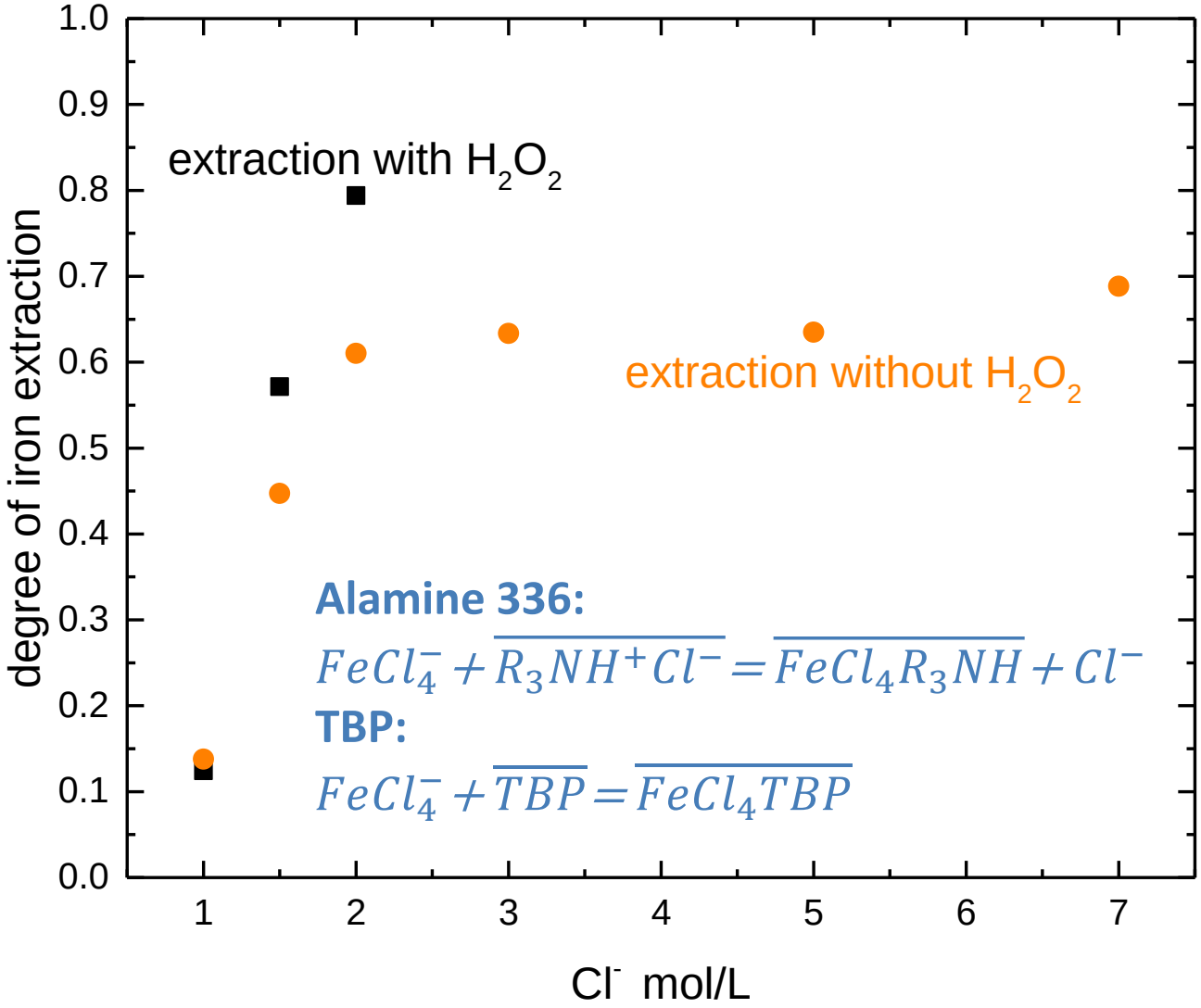
Fe extraction with different extractant concentrations and with H_2O_2



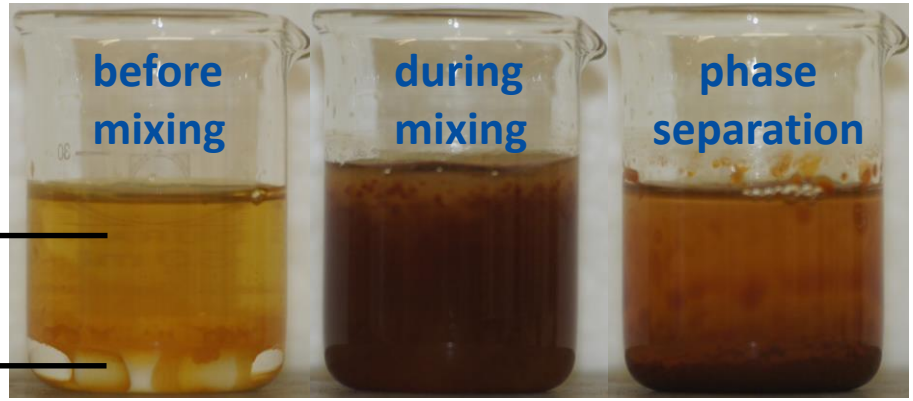
Fe extraction extraction with and without H_2O_2



reactive extraction of Fe with A336 + TBP

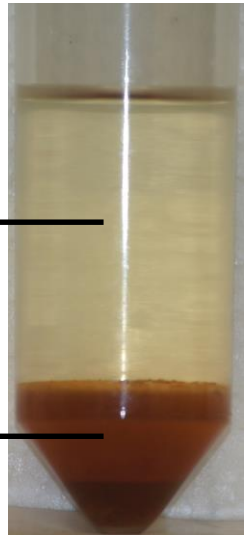


re-generation of solvent



organic solvent + metals

aqueous phase (NH_3 + NH_3HCO_3)

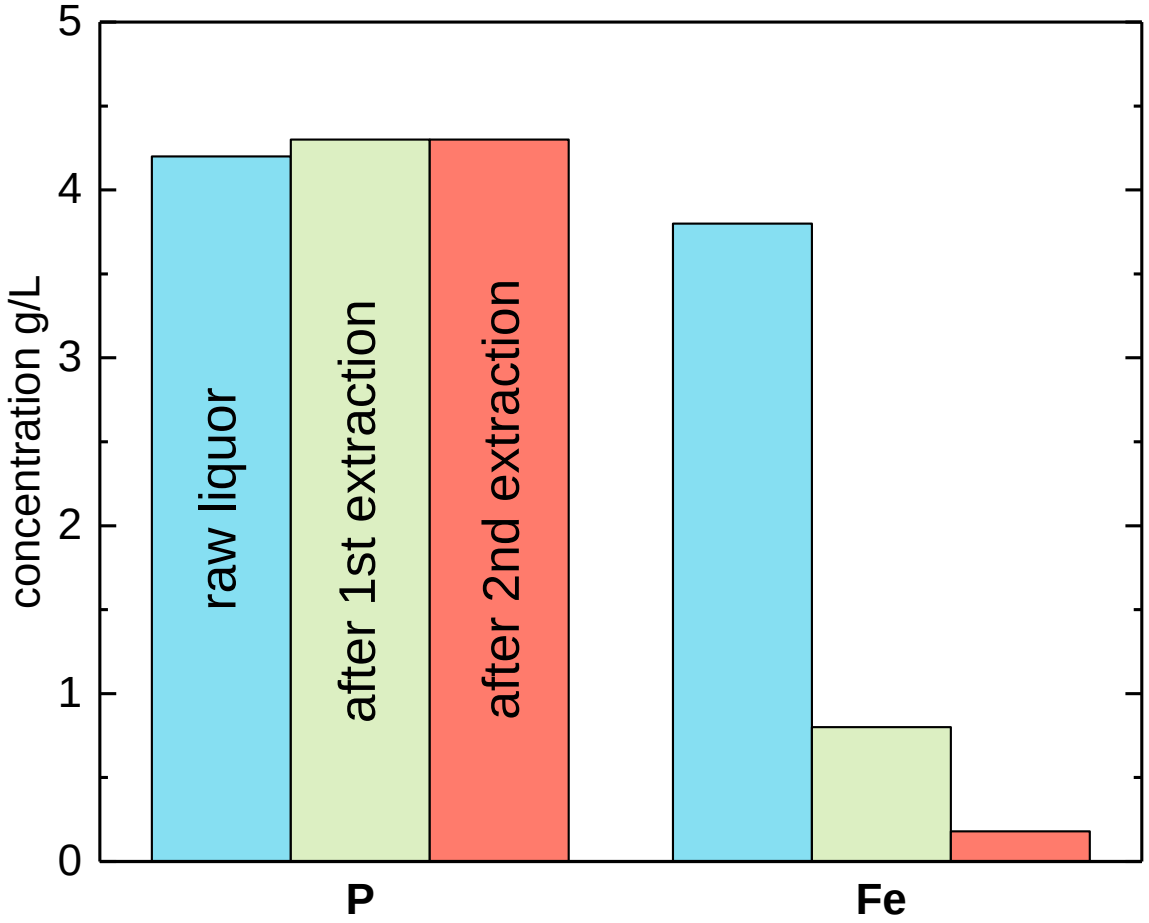


regenerated organic solvent

aqueous phase + metal precipitates

after centrifugation

2 stage extraction before precipitation of product



PULSE product

leaching of dried sludge

↓

filtration

↓

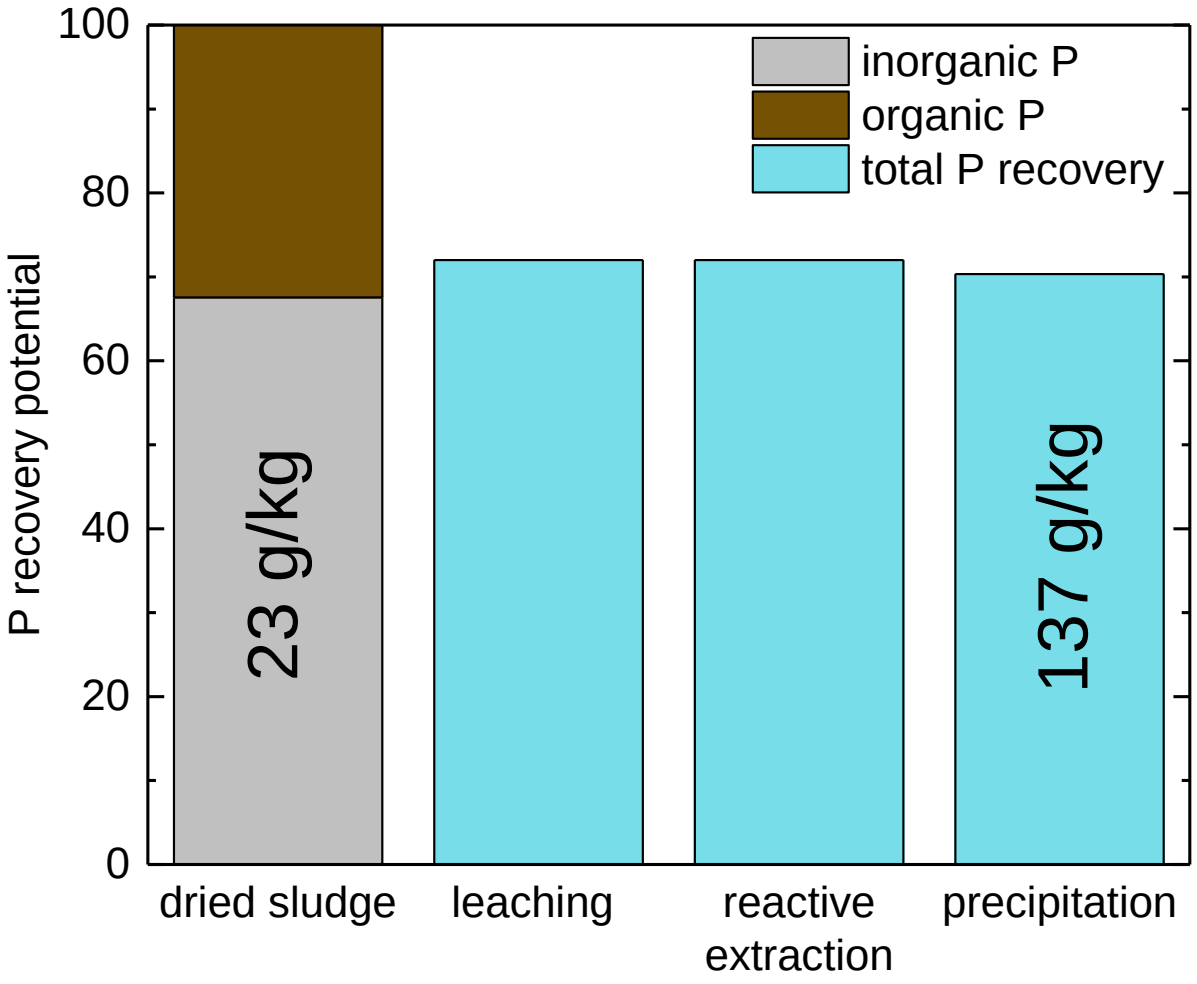
metal removal by reactive extraction

↓

CaP precipitation with NaOH (pH = 6 - 7)

Comp.	PULSE	Legislative limit
P_2O_5	29.2%	> 3 %
CaO	21.7 %	> 1.5 %
Total C	6 %	-
Al_2O_3	2.61 %	-
MgO	1.84 %	> 1.5 %
Fe	0.325 %	-
Mn	10800ppm	-
Cr	1530 ppm	-
Cu	294 ppm	600 ppm
Ni	51.6 ppm	120 ppm
Pb	38,8 ppm	150 ppm
Zn	8.97 ppm	-
Cd	0.735ppm	60 ppm
Hg	0.054 ppm	2 ppm

P recovery potential



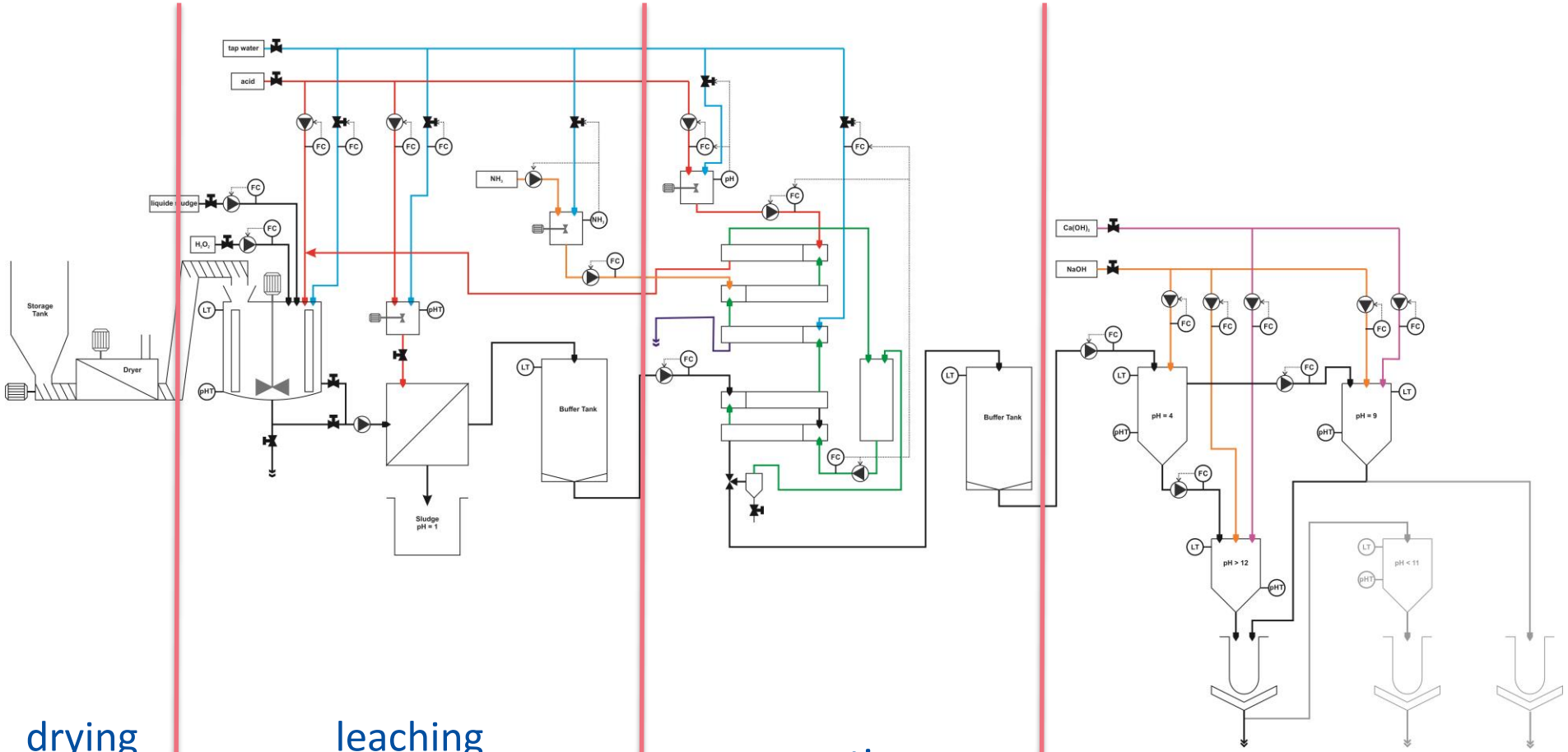
summary

- no significant difference between P leaching from wet or dry sludge
- P leaching depends only on pH and not on the type of acid
- extraction of metals at low pH only achieved with Alamine 336
- metals concentration in product is within the limits set by the EU fertilizer regulations except in case of Cr

further work

- lab scale optimization
- construction of demonstrator – 100kg/day of dewatered sludge capacity
- operation of demonstrator in Belgium, Germany, Ireland and Scotland
- LCA and scale up design

further work: PULSE demonstrator



drying + crushing

leaching + filtration

reactive extraction

precipitation + filtration

acknowledgements

- financing
  - Interreg North-West Europe
  - SPW Région Wallonne
- analytics
  - Faculty of Bioscience Engineering, Ghent University, Belgium
  - Prayon S.A., Belgium

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School of Life Sciences

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- Kimo C. van Dijk, Jan Peter Lesschen, Oene Oenema, 2016. Phosphorus flows and balances of the European Union Member States, Science of The Total Environment, Volume 542, Part B

- http://www.nweurope.eu/projects/project-search/phos4you-phosphorus-recovery-from-waste-water-for-your-life/

- Doetsch, P., Pinnekamp, Johannes, Montag, D., Rath, W., Grömping, M., 2010. Rückgewinnung von Pflanzennährstoffen, insbesondere Phosphor aus der Asche von Klärschlamm (Abschlussbericht PASCH). Institut für Siedlungswasserwirtschaft der RWTH Aachen, Aachen.

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- EC, 2019. Proposal for a REGULATION OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL laying down rules on the making available on the market of CE marked fertilizing products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009.