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## 1. Introduction

The distribution of phosphorus (P) resources is disproportionate and there is even increasing interest for P-based fertilizers. This has prompted many countries to promote circular P economy. Sewage sludge is a promising source for P recovery. During P recovery by sludge leaching, the metals are also leached and recycled which may be detrimental when the recovered product is used as a fertilizer. With the PULSE (Phosphorus University of Liège Sludge Extraction) process shown in Figure 1, it is possible to recover P from sludge and also remove metals during the recovery. P is recovered as calcium phosphate in the final product which can be used as fertilizer. It is also possible to modify the process to obtain P as magnesium salt.

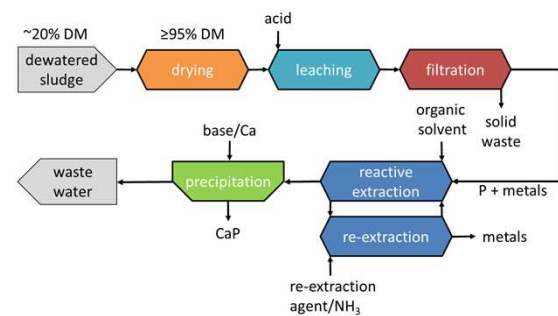


Figure 1: PULSE process (own illustration)

## 2. PULSE process development

During process development, different process options were evaluated and the most optimal option was selected using the cascaded option trees [1] as shown in figure 2. As the composition of sludge varies, it is imperative to understand reactions occurring as a function of pH in the different unit operations of the process to optimize and adapt the process parameters to the input sludge. Therefore, a MATLAB tool for simulating solid-liquid-liquid equilibria (SLL) was developed. With the help of this tool, it is possible to optimize the process for different types of sludge during pilot trials while minimizing experiments. The SLL tool can also be used to fit thermodynamic parameters such as stoichiometry and equilibrium constants required for simulations as shown in figure 3.

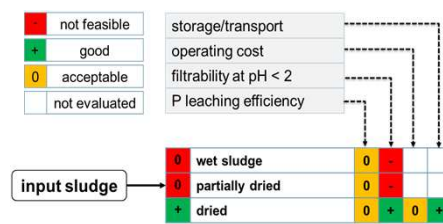


Figure 2: Cascaded option tree for sludge-input options [2]

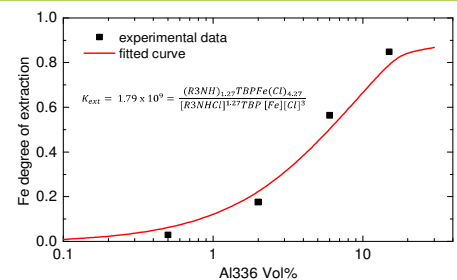


Figure 3: Fitting of thermodynamic parameters and simulation of solvent extraction of Fe [2]

## 3. P-rich PULSE product from demonstrator trials

The PULSE demonstrator as shown in figure 4 was operated with sludge from Belgium, Germany and Scotland. A P-rich product containing calcium phosphate with low metal content was obtained. For example, about 14 kg of dried product with a P<sub>2</sub>O<sub>5</sub> content of 27.4 % was obtained from 78 kg of dry German sludge during the trial. Granulation trials on the PULSE product carried out by Prayon S.A. showed that the product was well granulated as shown in figure 5.



Figure 4: (A) mobile PULSE demonstrator (© Uliège-Michel Houet); (B) drying, leaching & precipitation modules (© Uliège-Z. Shariff); (C) mixer-settlers for solvent extraction (© Uliège-Michel Houet)

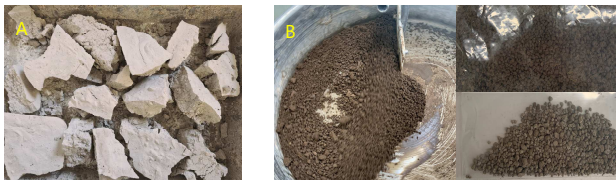


Figure 5: (A) P-rich PULSE product obtained from German sludge (© Uliège-Z. Shariff); (B) granulation tests of PULSE product (© Prayon S.A.)

## 4. Discussion and conclusions

- Compared to dewatered sludge, the use of dried sludge (DM < 90 %) results in lower acid consumption and also the filtration of solids after leaching is easy to achieve even at very low pH ~ 0.
- Hydrochloric acid was chosen for leaching because it is the cheapest of the inorganic acids and also gives better results in solvent extraction of metals. An example of P-leaching is shown in Figure 6.
- The metals Fe, Cd, Cu, Hg, Pb and Zn were extracted during solvent extraction while the change in P concentration of the leach liquor was negligible. Example of metals extracted is shown in figure 7.
- The overall P recovery in the pilot trials with German and Belgian sludge was 60 to 70%.

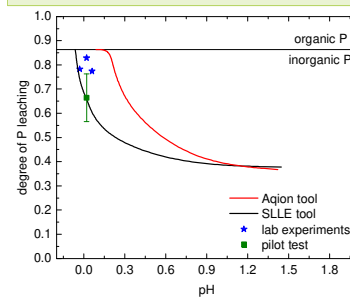


Figure 6: P leaching efficiency from dried digested German sludge with HCl [2]

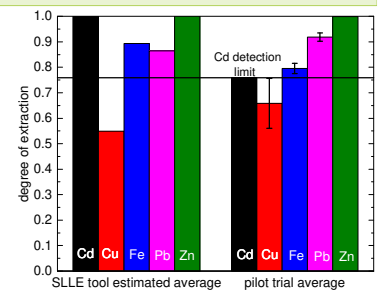


Figure 7: Metals extracted from German sludge liquor in 2 counter-current stages [2]