Recovery of phosphorus from dried sewage sludge and subsequent purification using reactive extraction

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Abstract: A new PULSE process for recovery of phosphorous from dried sewage sludge has been developed. In the PULSE process, acidic leaching is used to dissolve P from dried sludge. By using dried sludge, the consumption of acid for leaching is reduced and the separation of the solid and liquid fractions even at very low pH is easier as compared to using dewatered sludge. Metals and heavy metals that are co-leached with P are then removed by a reactive-extraction step. P can finally be precipitated as calcium or magnesium phosphate. The cascaded option-tree methodology has been used for evaluation of different process options and to develop the PULSE process. For gaining process understanding and process optimization an equilibrium modelling tool has been developed in MATLAB. In the experimental studies, the effect of different acids and pH on the leaching of P from dried sludge and different extractants for the subsequent metal removal have been investigated. Based on these lab-scale experiments, a pilot-plant scale demonstrator has been designed and built. Product samples containing calcium phosphate salts with a P_2O_5 concentration of up to 29 % have been produced.

Keywords: phosphorus; sludge drying; metal extraction; process development; equilibrium modelling

Introduction

Acidic leaching of sewage sludge or ashes is a common method used for recovering phosphorus (P), however acidic leaching also results in dissolution of metals. The limits for the concentration of metals in the sludge or fertilizers that can be applied to ground are continuously getting more stringent in EU and around the world. Therefore, the removal of metals during P recycling from sludge or ashes with high metal concentrations is essential. Reactive extraction is one of the options for separation of metals during P recycling. Considering the regional sludge processing and disposal techniques, the PULSE (Phosphorus ULiège Sludge Extraction) process has been developed to recover P from dried sewage sludge which includes the solvent extraction step for metal removal. The PULSE process is a modification of the PASCH process developed at RWTH Aachen (Doetsch, et al., 2010)**Error! Reference source not found.**.

Material and Methods

Lab-scale experiments were carried out for the different unit operations of the PULSE process i.e. drying, leaching, reactive extraction, precipitation and filtration. The different process options have been systematically varied guided by cascaded option trees to evaluate and select the best process options (Bednarz, Rüngeler, & Pfennig, 2014). In case of leaching, the effect of type of acids, pH, addition of oxidizer, temperature, and residence time was investigated. For reactive extraction, parameters such as concentration of extractants, pH, and phase ratio were studied. The effect of pH on P precipitation using different bases was also evaluated. Most of the experiments at lab scale were performed using dewatered undigested sludge from the Oupeye waste-water treatment plant (wwtp) near Liège (Belgium). The sludge was subjected to drying on the day of collection and crushed to a particle size of 5 mm or less. The chemical unit operations i.e. leaching, solvent extraction and precipitation are inter-dependent and the optimal process conditions depend on the detailed sludge composition. To be able to optimize the process for each specific sludge, a MATLAB tool for computing the concentrations of all involved species at equilibrium was developed. The equilibria include the solid-liquid equilibrium for leaching and precipitation, the speciation in the aqueous phase, as well as that with the reactive organic phase in reactive extraction as a function e.g. of pH and other process parameters (Shariff, Fraikin, Léonard, & Pfennig, 2020).

Results and Discussion

There was no significant difference in the P leaching efficiency of dried and dewatered sludge which was found to be 69.1 % and 72.6 % respectively at a pH of about 0.5 for leaching with HCl. Half or even lesser amount of water is required to fluidize dried sludge as compared to the dewatered sludge. Filtration of dried sludge is easier as compared to dewatered sludge. The P leaching efficiency was not affected by the type of acid used and was only dependent on the pH as shown in Figure 1. It was also observed that mostly inorganic P and only a small fraction of organic P was leached under acidic conditions.

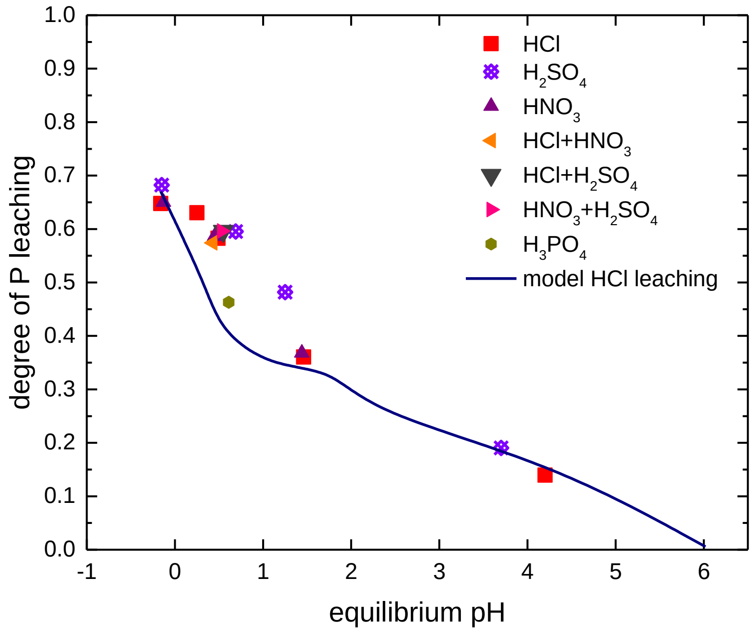

Figure 1: P-leaching efficiency from dry sludge. Sludge: Oupeye wwtp, S/L ratio: 0.25, leaching duration: 60 min, DM: >97 %, room temperature

Among the different solvents studied as shown in Figure 2, the solvent system containing Alamine 336, tri-butyl phosphate, Exxal10 (modifier) dissolved in Ketrul D80 (Kerosene) was chosen for the PULSE process. Metals such as As, Al, Cr and Ni were not extracted. The solvent can be efficiently regenerated by using alkaline solutions as shown in the Figure 3. The composition of the precipitated final product obtained from purified (after 2 stages of metal extraction) leach liquor by raising the pH around 7 to 8 using sodium hydroxide as base is shown in Figure 4. The concentration of Cr was particularly high, which was traced back to the atypically high Cr concentration in the inflow to the Oupeye wwtp plant.

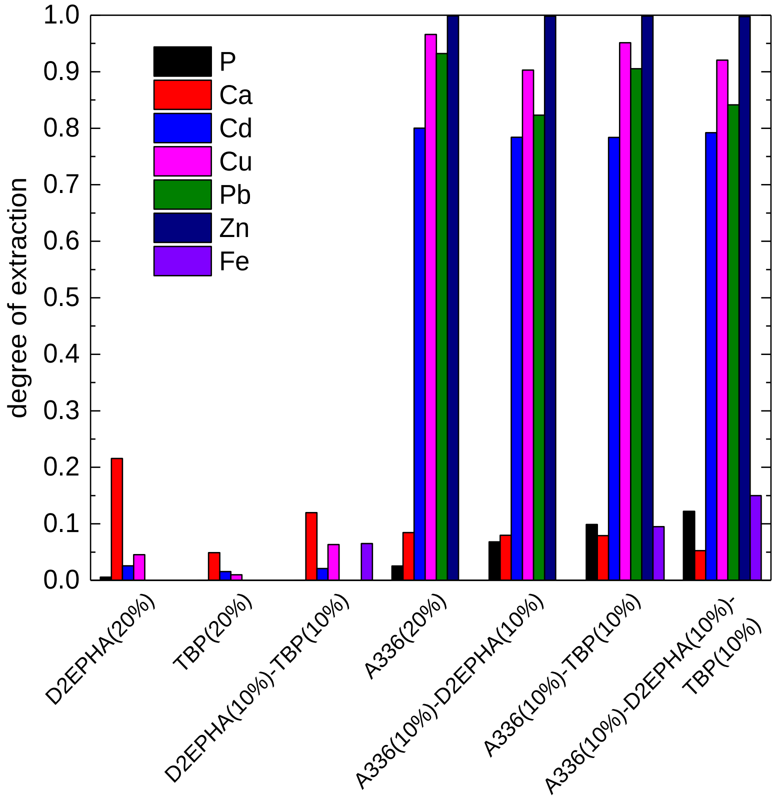

Figure 2: Extraction efficiency of metal from HCl leach liquor obtained at 1 mol/l , diluent: Ketrul-D80, modifier for Alamine336-Exxal10, solvent concentration indicated in vol%, O/A phase ratio: 2

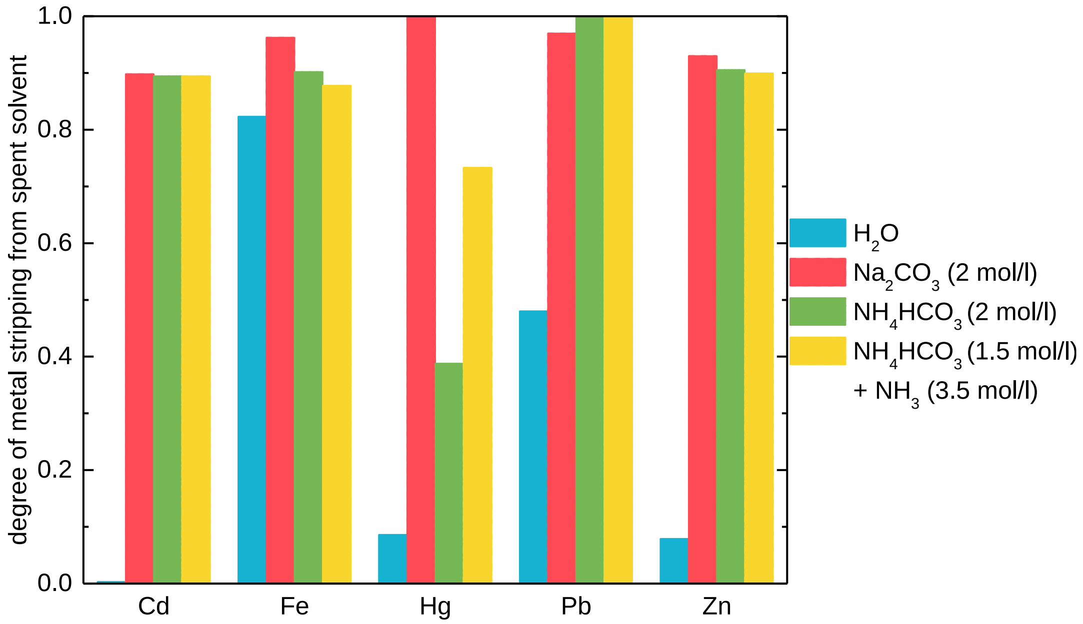

Figure 3: Degree of re-extraction of metals from spent extractant. Spent extractant: 10 vol% Alamine 336 + 10 vol% TBP + 3 vol% Exxal 10 + 77 vol% Ketrul D80, O/A phase ratio: 1

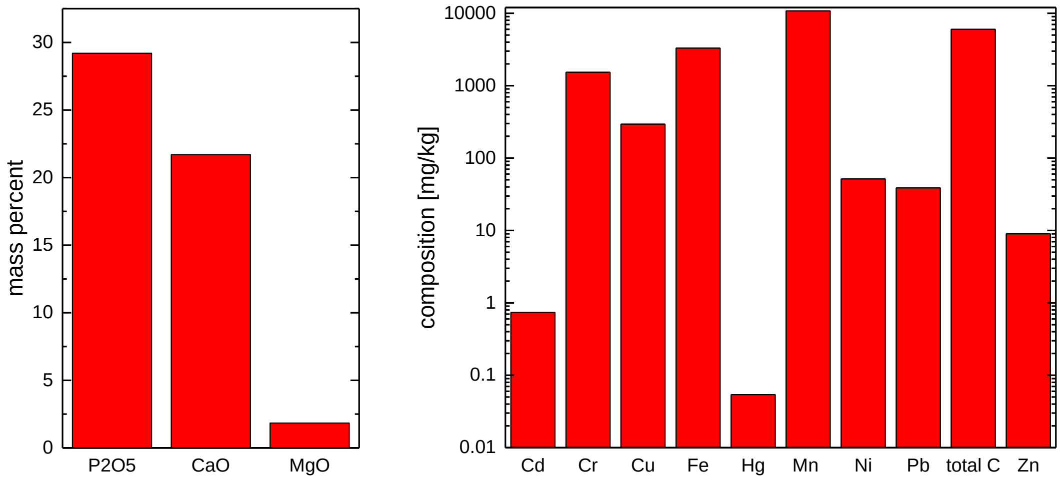

Figure 4: Composition of the product obtained from PULSE process at lab scale

Conclusions

Sludge drying doesn't have significant effect on P leaching efficiency. But dried sludge consumes less acid for leaching and also facilitates easier filtration of the of the slurry after leaching. Chemical leaching is strongly dependent on pH and mostly dissolves inorganic P. The optimum pH for P leaching depends on the metals, their oxidation states and concentrations. Reactive extraction provides high degree of extraction for a number of metals, making it possible to recover P from metal-rich sludge without compromising the quality of the final product.

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