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PHOSPHORUS RECOVERY BY USE OF SLUDGE FRACTIONATION

Fosforutvinning genom slamfraktionering

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Abstract

In working towards a sustainable society, recycling and recovery of products together with handling of scarce resources must be considered. The growing quantities of sludge from wastewater treatment plants and the increasingly stringent restrictions on landfilling and on agricultural use of sludge are promoting other disposal alternatives. Sludge fractionation, providing sludge volume reduction, product recovery and separation of toxic substances into a small stream, has gained particular interest. The potential for phosphate release and recovery from treated sewage sludge was investigated in Kristina Stark's PhD-thesis as an alternative for agricultural use in urban areas. Leaching and recovery experiments were performed on different sludge residues.

Results showed that acid or alkaline leaching is a promising method to release phosphate from sewage sludge treated with supercritical water oxidation, incineration, or drying at 300°C. The leaching is affected by a number of factors, including how the sludge residue has been produced, the origin of the sludge residue, the quantity of chemicals added and the presence of ions in the leachate.

The results may be beneficial for minimizing the use and cost of chemicals, and give increased knowledge for further development of technology for phosphate recovery.

Key words – Ash, phosphorus release, phosphorous recovery, sludge fractionation, supercritical water oxidation, sustainable sludge handling

Sammanfattning

Vägen till det uthålliga samhället går via skapande av kretslopp och utvinning av produkter tillsammans med resurshushållning. De växande slammängderna från avloppsreningsverk, fler restriktioner för deponi och jordbruksanvändning öppnar för andra alternativ av slamomhändertaganden. Speciellt slamfraktionering har visats stort intresse eftersom den möjliggör minskad slammängd, produktutvinning och separering av toxiska substanser i en liten delfraktion. Potentialen av fosforfrigöring och utvinning från behandlat avloppsslam har undersökts i Kristina Starks doktorsavhandling. Förutsättningen har varit att jordbruksanvändning inte är möjlig i den urbana staden. Laknings- och utvinningsförsök har genomförts med slamrest från superkritisk vattenoxidation (SCWO), aska från förbränning och torkat slam vid olika temperaturer.

Resultaten visar att lakning med syra eller bas är en lovande metod att frigöra fosfat från avloppsslam behandlat med SCWO, förbränning eller torkning vid 300°C. Lakningsresultaten påverkas av flera faktorer, såsom dess slamsammansättning, slambehandlingsmetod, tillsatt kemikaliemängd, närvaro av joner i lakningsvätskan.

Resultaten kan vara användbara för att minska användning och kostnad för kemikalier samt ge ökad kunskap om fosforutvinning.

Introduction

Sewage sludge as a resource

One crucial factor for traditional wastewater handling in central systems today is the difficulty in recycling resources in a reliable way. Agricultural use is often regarded as the best alternative if the pollutants in the sludge are below limiting and guidance values. However, a lack of acceptance from the food industry and the public is preventing widespread adoption of this option (Hultman et al., 2001a; Bengtsson and Tillman, 2004).

Landfilling, land application and incineration methods are currently the dominant modes of disposing of the sludge from urban wastewater treatment plants

(WWTP) (Svanström et al., 2004). In Swedish WWTPs, the most common option is land application (remediation of soils, parks, golf courses) (Stark, 2005a).

Sludge handling in Sweden has so far been regarded as a disposal problem (Bengtsson and Tillman, 2004), but the sewage sludge may be regarded not only as a threat to the environment but also as a resource. In sustainable sludge handling, resources are efficiently recycled without flows of harmful substances to humans or the environment (Harremoës, 1996). Sewage sludge has the potential to be used as a resource in a variety of options as for internal and external purposes in the WWTP (Morse et al., 1998; Hultman et al., 2001b; Stark, 2002a). The organic material may be used for soil conditioning, energy production, adsorption material or production of organic compounds as organic acids. The inorganic material may be used for reuse as precipitation chemicals, use in building materials and possible recovery of valuable metals. Nutrients, such as phosphorus, nitrogen and potassium, are also resources found in the sludge. Special attention may be given to the non-renewable phosphorus.

Possibilities for phosphorus recovery

Phosphate may be recovered from different sources (urine, wastewater, supernatant, sewage sludge) by direct use or by different technologies (Crystalactor, PhoStrip, KREPRO, BioCon, Aqua Reci). There has been increasing interest in sludge fractionation, which includes sludge volume reduction, product recovery and separation of toxic substances into a small stream. Different options for sludge fractionation are summarized in Table 1.

Methods and Materials

The overall aims of the thesis were to investigate the potential for phosphate release and recovery from treated sewage sludge. The intention was to study solutions for a large WWTP within the long-term goal of phosphorus recovery when agricultural use is not possible in an urban area. Experiments were performed with sludge residue from supercritical water oxidation (SCWO), dried sludge and ash from incineration, followed by evaluations of leaching and recovery. Some of the main questions investigated were:

- What are the respective advantages and disadvantages of acid and alkaline leaching?
- How do differences in sludge residues affect phosphate release and recovery?
- How do different parameters affect the result (temperature, pH, etc.)?
- What degree of recovery can be expected from certain types of sludge?

The experiments were all conducted on laboratory scale at the Water Chemistry laboratory at the Department of Land and Water Resources Engineering, KTH, Stockholm. The materials investigated were sludge residues from the SCWO process, ash from incineration and dried sludge. The studies were divided mainly into acid, alkaline leaching and recovery experiments. The experimental set-up and procedure are presented in Figures 1 and 2. The development of the experimental procedure for estimation of phosphorus release and recovery is described in detail in Stark (2002c); Stark and Hultman (2003); Stark (2005b); Stark et al. (2005c), respectively, together with the methods of analysis used.

Table 1. *Different options for sludge fractionation (Hultman et al., 2001b; Stark, 2002a; Saktaywin et al., 2005).*

Methods	General purpose
Physical	
Heat/pressure	Solution of sludge components, sludge conditioning
Mechanical	
Mechanical devices, ultrasonic	Disruption of cells for improved sludge degradation
Biological	
Enzymes	Solution of sludge components, increased biodegradability of the sludge
Anaerobic treatment	Production of organic acids, release of phosphates
Sulphate reducing bacteria	Production of sulphides for release of phosphates and precipitation of metals
Sulphur, sulphide and ferrous oxidation bacteria	Production of hydrogen ions for release of metals from sludge
Chemical	
Acids, bases, oxidising agents (ozone, hydrogen peroxide, etc.)	Hydrolysis of sludge, release of different sludge components, conditioning of sludge, increased biodegradability of sludge
Complexing agents	Release of metals, etc. from sludge

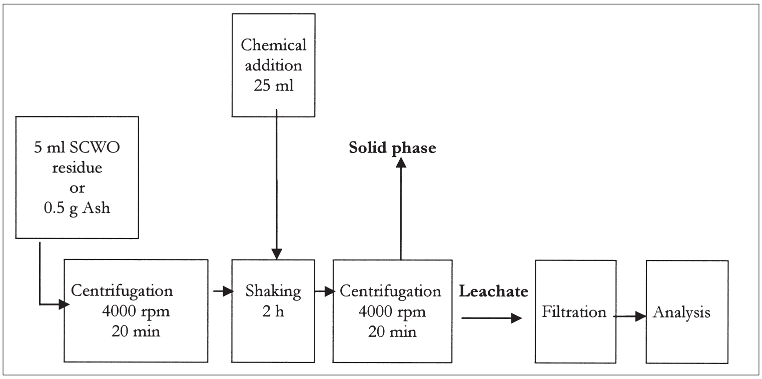

Figure 1. *The procedure used for leaching from SCWO residue or ash at different molar concentrations.*

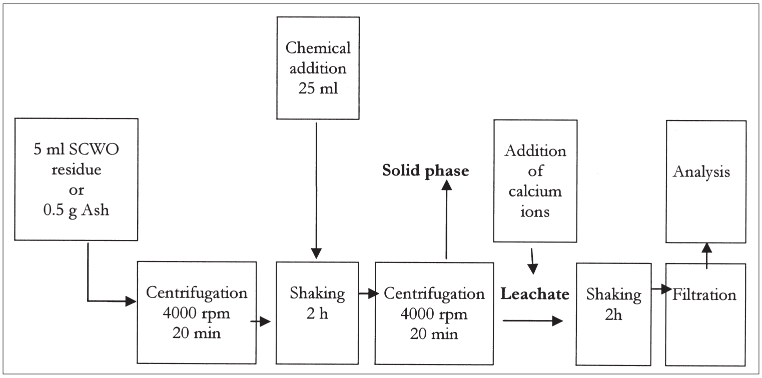

Figure 2. *The procedure used for recovery from SCWO residue or ash.*

Results and Discussion

Factors affecting phosphorus leaching

Some of the behaviours observed in the experiments performed (Stark, 2005b) and possible explanations for these are discussed and compared with results in the literature.

Different added amounts of acid or base

The results of the experiments performed showed that phosphate was leached more easily with the acid (hydrochloric acid) at room temperature than with the base (sodium hydroxide), in accordance with literature, e.g. Koutsoukos and Valsami-Jones (2004). The largest degree of released phosphate, 80–100 % at acid concentrations of 0.1M HCl, was obtained by leaching SCWO residue. Alkaline leaching of SCWO-residue at 1M NaOH gave 50–70 % released phosphate (Figure 3). Acid leaching of ash at1M HCl gave 75–90 % released phosphate, while alkaline leaching of ash at 1M NaOH gave 40–70 % released phosphate (Figure 4). The results showed that both added acid and base were sparingly consumed for leaching different components, thus, the

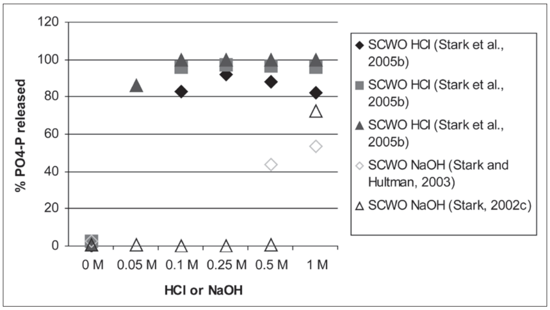

Figure 3. *Phosphate released from SCWO residue by acid or alkaline leaching.*

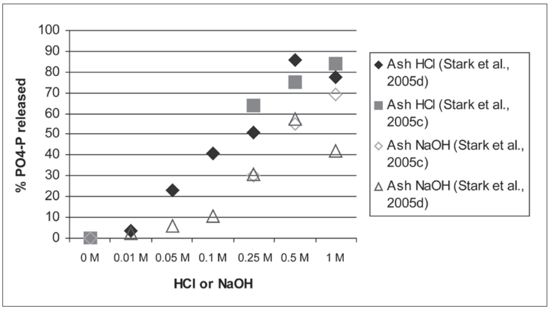

Figure 4. *Phosphate released from ash by acid or alkaline leaching.*

Table 2. *Components leached in different studies on ash and SCWO residue.*

	PO_4 (%)	Al (%)	Fe (%)	Ca (%)	References
ASH					
1M HCl					
Al, digested	87	23	6	83	Stark et al., 2005c
Al, digested	77	–	–	–	Stark et al., 2005d
1M H2SO_4					
Al, raw sludge	88	65	32	5.6	Schaum et al., 2004
Fe, raw sludge	96	71	16	6	Schaum et al., 2004
Fe, digested	94	70	21	4.3	Schaum et al., 2004
EBPR*	100	84	19	8	Schaum et al., 2004
Al, digested	0.05	–	–	–	Stark et al., 2005d
1M NaOH					
Al, raw sludge	31	31	–	–	Schaum et al., 2004
Fe, raw sludge	28	31	–	–	Schaum et al., 2004
Fe, digested	3	44	–	–	Schaum et al., 2004
EBPR	54	40	–	–	Schaum et al., 2004
Al, digested	70	9.5	0.04	0.19	Stark et al., 2005c
Al, digested	42	–	–	–	Stark et al., 2005d
SCWO residue					
1M HCl					
Fe, digested	100	–	2.80	–	Stark et al., 2005c
Fe, digested	82	–	5.3	–	Stark, 2002c
Fe, digested	95	–	18	–	Stark, 2002c
Fe, digested	48	–	–	–	Stark et al., 2005d
HCl pH below 2					
Fe, digested	76	92	10	86	Stendahl, 2005
H_2SO_4 pH below 2					
Fe, digested	84	100	12	17	Stendahl, 2005
1M H_2SO_4					
Fe, digested	0.05	–	–	–	Stark et al., 2005d
1M NaOH					
Fe, digested	53	43	0.07	–	Stark et al., 2005c
Fe, digested	74	–	0.045	–	Stark, 2002c
Fe, digested	13	–	–	–	Stark et al., 2005d

data indicated that acid or base were added in surplus. A higher efficiency of consumed chemicals and released phosphate can be obtained by performing experiments under pH control (Stark, 2005b). This corresponds to other studies showing a high release at low pH value (Hansen et al., 2000; Schaum et al., 2004).

Sludge residue composition

The composition of the sludge residue affects the leaching results and, thus, the recovery. The optimal situation for phosphate recovery is to obtain a high release of phosphate and a low release of heavy metals and metals that later on can give rise to separation problems.

Table 2 shows the behaviour of PO_4, Al, Fe and Ca in different leaching studies on ash and SCWO residue (Schaum et al., 2004; Stendahl, 2005; Stark 2002c; Stark et al., 2005c; Stark et al., 2005d;). The different chemicals added, the precipitation agent in WWTP and the sludge used in the process are given. The leaching took place at room temperature. Phosphate released in these experiments is in accordance with other studies published. Unfortunately, all leached ions were not analysed and in most cases only iron and phosphate were analysed. As can be seen from Table 2, in most cases Al was released together with the phosphate in acid leaching, while iron had a lower release. Alkaline leaching also showed a release of Al and PO_4, while Ca and Fe showed a low release. After the thermal treatment, iron is probably present as iron hydroxide, which is difficult to break. The leaching of iron was higher from ash than from SCWO residue. This may be explained by the higher temperature in the incineration than in the SCWO process. There was no significant difference in phosphate release between ash and SCWO residue at this leaching concentration (1M). However, a lower acid concentration has shown a better release from SCWO than from ash (Stark et al., 2005c and d). The experiments in Stark et al., 2005d showed a low release of phosphate from SCWO residue compared with the other studies (Stark, 2002c; Stark and Hultman, 2003). This is probably depending on a long storage time of the residue (about 3 years) before the leaching experiments (Figure 5). This may explain the differences in results from earlier studies comparing SCWO and incineration. The metal/phosphorus ratio was higher with acid than with base (Stark, 2002c; Stark et al., 2005c). This means alkaline leaching will preferentially dissolve phosphorus with a lower metal contamination compared to acid leaching and is in accordance with results presented by Cheeseman et al. (2003) and Stendahl and Jäfverström (2004).

Temperature

In many cases, higher temperatures increase the reaction process. However, this seems not to be favourable in phosphate leaching, as shown by the results from Stark (2002b), where the experiments performed at 20°C (Stark, 2002c) were compared with leaching studies performed at 90°C (Stendahl, 2001). The results show that iron was released differently with acid leaching at 20°C and 90°C (Figure 6 and 7). At room temperature, 100 % of the phosphate was released at 0.1M HCl and only 1 % iron, while at 90°C, 64 % phosphate was released and 71 % iron. A possible reason is that the metal bonds break at different rates and dissolution is therefore likely to be a multi-tier process, the overall rate of which will be defined by the slowest breaking bonds (Koutsoukos and Valsami-Jones, 2004). The higher temperature together with acid dissolved the strong iron precipitates and caused precipitation of phosphate with dissolved ions. In the case of alkaline leaching, the release was similar at both temperatures, with a low iron release. This may be due to the high concentration of hydroxide ions and to the fact that $FeOH_3$ is difficult to dissolve.

Optimising leaching and recovery

One possibility to prevent the mixture of leached components is to have a pre-treatment step. This would include pre-washing of the sludge residue, followed by acid leaching and alkaline leaching. With pre-washing, soluble matter that would otherwise consume chemicals can be removed. The first step could also be an acid leaching step. The majority of calcium and magnesium compounds may be removed at slightly acid conditions. Some phosphate is also released during this acidic leaching and conditions should be chosen where the release of

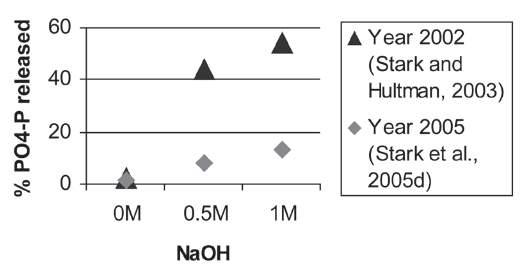

Figure 5. *Phosphate release from fresh and stored SCWO residue.*

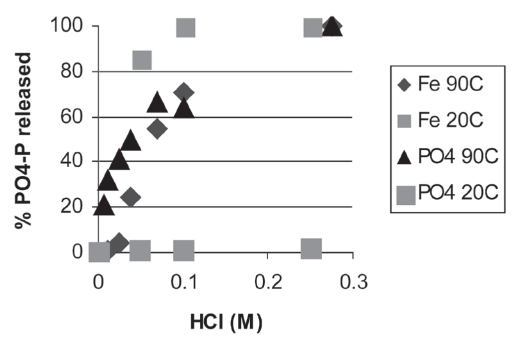

Figure 6. *Comparison of released phosphate and iron at 20°C and 90°C with acid leaching (modified from Stark, 2002b).*

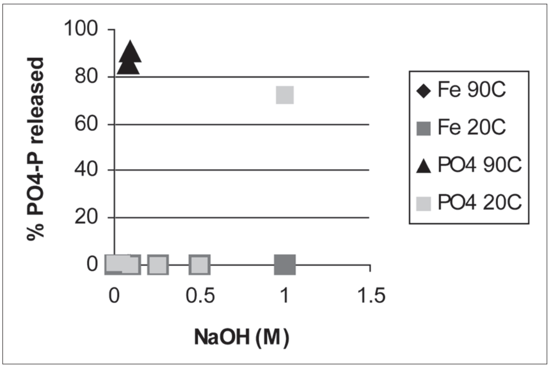

Figure 7. *Comparison of released phosphate and iron at 20°C and 90°C with alkaline leaching (modified from Stark, 2002b).*

iron and heavy metals is low. This stream may be combined with supernatant from dewatering of digested sludge or side-streams from processes like Pho-Strip to recover part of the phosphorus supplied to the wastewater treatment plant (Stark, 2002b).

Costs

Phosphorus recovery can be seen as technically possible, but the economic feasibility of phosphorus recovery from sewage can still be regarded as dubious (Roeleveld et al., 2004). In Balmér et al. (2002), the cost analysis of six potential phosphorus recovery systems was compared. The most favourable system, apart from direct agricultural application, was the advanced technologies (PhoStrip, KREPRO and BioCon) that showed less cost to the reference alternative (incineration). The increased cost was about 1.5–5 Euro/person or 3.5–9 Euro/kg P. This would mean an increase of 1–3 % in Swedish water and wastewater taxes. The leaching and recovery step are not involving the greatest cost. Instead, the largest investment required is in the technology for sludge treatment. In Stark et al. (2005a), the economic aspect is discussed, since phosphate recovery cannot be profitable without also taking into account the savings in the WWTP. In Stark et al. (2005a) and Stark et al. (2005d), total cost for technologies and estimations of costs for leaching phosphate from ash or SCWO residue are described and show about the same cost for the both processes. This is also presented by Levlin et al. (2005), namely the leaching cost was estimated to be 7 Euro/kg P for the ash and 7.2 Euro/kg P for the SCWO when all matter was leached. Leaching only phosphate, calcium and aluminium gave a cost of 0.32–1.43 Euro/kg P from incinerated ash or SCWO residue.

Future urban wastewater systems with recovery

Local, central and regional solutions

Improved sustainability of the urban infrastructure may be achieved with the help of technical arrangements and suitable management systems. The direction of development is likely to include both improving the existing WWTP in a more sustainable way and introducing source separation technologies to a larger extent. It is probable that future urban wastewater systems will include local, central and regional solutions that have in common resource conservation and production of usable products.

In sustainable sludge handling, the sludge will have to be utilised as a raw material to produce different useful products. One scenario is that the WWTP will consist of a sludge treatment process followed by a recovery unit and, depending on scale and size, treatment will be central or regional. In Sweden, the larger cities may have a particular treatment technology installed, such as SCWO or incineration, and may be able to receive sludge from surrounding towns.

Conclusions

Phosphorus recovery is not justified only based on market economy. However, the concern of phosphorus as a non-renewable resource and diffuse disposal of phosphorus-rich sludge, residue or ash may justify phosphorus recovery based on environmental concerns. Other important aspects are the secondary effects of phosphorus recovery including less sludge amounts to handle and recovery of other components such as precipitation agents.

What are the respective advantages and disadvantages of acid and alkaline leaching?

The choice of using acid or alkaline leaching is depending on the desired achievement. Acid leaching is advantageous in respect of high phosphate release and high sludge reduction, while alkaline leaching preferentially gives lower metal contamination of the leachate. The result in the thesis showed phosphate was leached more easily with the acid than with the base. The largest degree of leached phosphate was obtained by leaching SCWO residue with acid (80–100 % at acid concentrations 0.1M HCl). Acid leaching of sludge incineration ash gave 75–90 % released phosphate at the concentration 1M HCl. Alkaline leaching of SCWO residue gave 50–70 % leached phosphate at the concentration 1M NaOH, while 40–70 % was obtained from the ash. Both acid and alkaline leaching showed that iron was not released simultaneously with phosphate, which facilitates the further recovery step.

How do differences in sludge residues affect phosphate release and recovery?

Prediction of phosphate release is a complex process, as the components leached from the sludge residue depend on its original composition and the presence of ions affects the phosphate release. Leached iron and aluminium ions together with phosphate give rise to separation problems in obtaining a phosphorus product. The type of bonds of phosphorus and metals during incineration and especially SCWO still need to be further studied.

The results showed that it was easier to release phosphate from the SCWO residue than from the ash at low acid concentrations (0.05M–0.5M HCl). The different behaviour for the SCWO process could be due to that the SCWO residue may be more reactive due to smaller particle size than the ash. It was found that pre-treatment of the ash may be important for better release of phosphate and storage of SCWO residue influenced the release of phosphate. The difference in composition of ash and sludge residue samples had no significant influence on release of phosphate at higher concentrations of acid (1M HCl). Better efficiency of release for any of the ash or SCWO residue samples at lower base concentration was not observed. Results for alkaline leaching showed higher release from ash at 1M NaOH than SCWO residue and dried sludge at 300°C.

How do different parameters affect the result of phosphate release (temperature, pH, etc.)?

The result in this study has shown that the temperature used in acid leaching of SCWO residue influences the relative release of ferric and phosphate ions. The leaching was favoured at room temperature compared to 90°C. A reason for this might be due to the metal bonds breaking at different rates and iron hydroxide may have been dissolved at higher temperature. The temperature of thermal treated digested sludge, incineration ash and SCWO residue influences the release of phosphate, aluminium and ferric ions. The dried sludge treated at higher temperature showed less release of phosphate. This may be related to the phenomena of glassification and crystallization that may occur with increasing temperature.

The results showed that both added acid and base were sparingly consumed for leaching different components, thus, the data indicated that acid or base were added in surplus. A higher leaching efficiency for phosphate was shown at experiments under pH control (values of pH below 3).

What degree of recovery can be expected from certain types of sludge?

The results showed that phosphate recovery as calcium phosphate was possible from both ash and SCWO residues. Any differences in implementation of a certain technology would depend on cost, environmental regulations and social aspects. Approximately 50 % of total phosphorus was recovered at 1M NaOH. Higher result may be reached with acid leaching.

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