RECOVERY OF PHOSPHATE FROM SEWAGE SLUDGE AND SEPARATION OF METALS BY ION EXCHANGE

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Abstract

Ion exchange can be used to separate phosphorus from iron when phosphorus is recovered from sewage sludge. A review of the use of ion exchange for phosphorus recovery is presented followed by a discussion on how to use ion exchange for separation of metal and phosphate. Example of processes for recovering of phosphate with ion exchange are BioCon and RemNut. In the BioCon-process is ash from sludge incineration leached with acid and the metal ions are separated from the phosphate with a ion exchange process, producing iron chloride and phosphoric acid. In the RemNut-process is phosphorus recovered from the effluent of the sewage treatment plant as magnesium ammonium phosphate. Phosphate, which without a ion exchange process is recovered as iron phosphate, has no commercial value as raw material for the phosphate industry. By mixing a cation exchange resin with the sludge, hydrogen ions from the ion exchanger can dissolve metal ions which are taken up by the ion exchanger. If a magnetic resin is used it can be separated from the sludge with a magnetic drum. Using the acid released from the ion exchange resin to leach the sludge decreases the consumption of chemicals needed for the process. Ion exchange textiles and ion selective membrane can also be used for phosphorus recovery processes.

Keywords

Ion exchange, phosphorus recovery, sewage sludge

BACKGROUND

A national goal has recently been proposed in a report to the Swedish government that at least 75% of phosphorus from wastewater should be recovered at latest 2010 without risks for health and environment (Wallgren, 2001). In the sewage treatment plant phosphorus is removed from the wastewater by precipitation with iron salt. However, since phosphorus is needed as a fertiliser in the agriculture, a requirement for getting sustainable wastewater treatment is to create method to use the phosphorus from the wastewater as a fertiliser in the agriculture. Most of the phosphorus used in the agriculture originates from mining of phosphate ores. The global deposits of economically mine able phosphate are estimated to be 10^9 ton phosphorus and the total amount phosphorus in the sediments is estimated to be 10^{15} ton (Butcher et al., 1994). Many different phosphate minerals are available, but only apatite (calcium phosphate, $Ca_3(PO_4)_2$) is used for phosphate production (Corbridge, 1995). Phosphate can be economically produced by leaching apatite mineral with sulphuric acid (McKetta and Cunningham 1990):

$$Ca_3(PO_4)_2 \text{ (s) } + 3\,H_2SO_4 + 3x\,H_2O \longrightarrow 2\,H_3PO_4 + 3\,CaSO_4 \cdot xH_2O \text{ (s)}$$
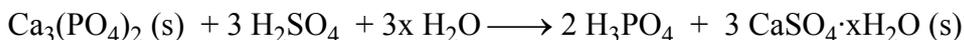
In 1995 the world phosphate rock production was 160 000 ton per year (as P_2O_5), having tripled over the last 40 years. About 90% of this amount is used as fertiliser. At this rate of consumption the known apatite reserves have been estimated to last for a period up to 1000 years. However, if the present increase in world population and the increasing need for fertiliser for food production is taken into account, the supply of phosphate may well be crucial within a century. Apatite ore is thereby a limited resource that must be preserved by phosphate recovery.

Phosphorus may be recovered from sewage sludge by leaching with acid (Levlin et al., 1998, 2000, Levlin, 1999). The solubility of phosphate compounds decreases with increasing pH-level. Phosphate compounds can depending on the pH-level be dissolved as:

$$MePO_4 \text{ (s)} + 3\,H^+ \longrightarrow H_3PO_4^{\,o} + Me^{3+} \qquad \text{(pH < 2.15)}$$

$$MePO_4 \text{ (s)} + 2\,H^+ \longrightarrow H_2PO_4^{\,-} + Me^{3+} \qquad \text{(pH > 2.15 and < 7.20)}$$

$$MePO_4 \text{ (s)} + H^+ \longrightarrow HPO_4^{\,2-} + Me^{3+} \qquad \text{(pH > 7.20 and < 12.02)}$$

$$MePO_4 \text{ (s)} \longrightarrow PO_4^{\,3-} + Me^{3+} \qquad \text{(pH > 12.02)}$$
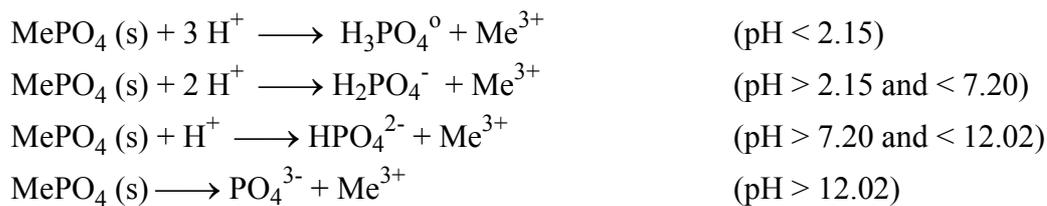
Two systems for phosphorus recovery from sludge in wastewater treatment plants with chemical precipitation with iron salts is at present considered, KREPRO (Hansen et al., 2000, Hagström et al., 1997) and Bio-Con which uses ion exchange processes. In the two systems the iron content in the sludge is dissolved by acid together with the phosphate. After dissolution, the leachate contains a mixture of different ions including iron, together with phosphoric acid, which must be separated in a further step.

In the KREPRO-process is the heavy metals precipitated with sulphide and the phosphate as ferric phosphate. Without removing the iron, phosphate will preferentially be precipitated as iron phosphate, which have a lower solubility than calcium phosphate. Iron phosphate has no commercial value as raw material for the phosphate industry, and the low solubility makes it less favourable to use as fertilizer. Since the phosphate in the sludge originate from phosphorus products produced from apatite ore, recovering the phosphate as iron phosphate will not preserve the limited apatite resources. Iron phosphate is a much more common mineral in the ground than apatite. Use of ion exchange processes as in the BioCon-process, make it possible to recover the phosphate as phosphoric acid, which is produced from apatite ore, thus preserving the limited apatite resources and the resources, mainly sulphur, needed for producing phosphoric acid from apatite.

ION EXCHANGE PROCESSES USED FOR PHOSPHORUS RECOVERY

The BioCon-process
The BioCon-process shown by figure 1 (Svensson, 2000), in which ion exchange is used for phosphorus recovery after leaching with acid, has been developed by a Danish company. In Sweden, the municipality of Falun intend to apply for a permit for sludge incineration with P-recovery from the incineration ash, and a plant using the BioCon-process is expected be built.
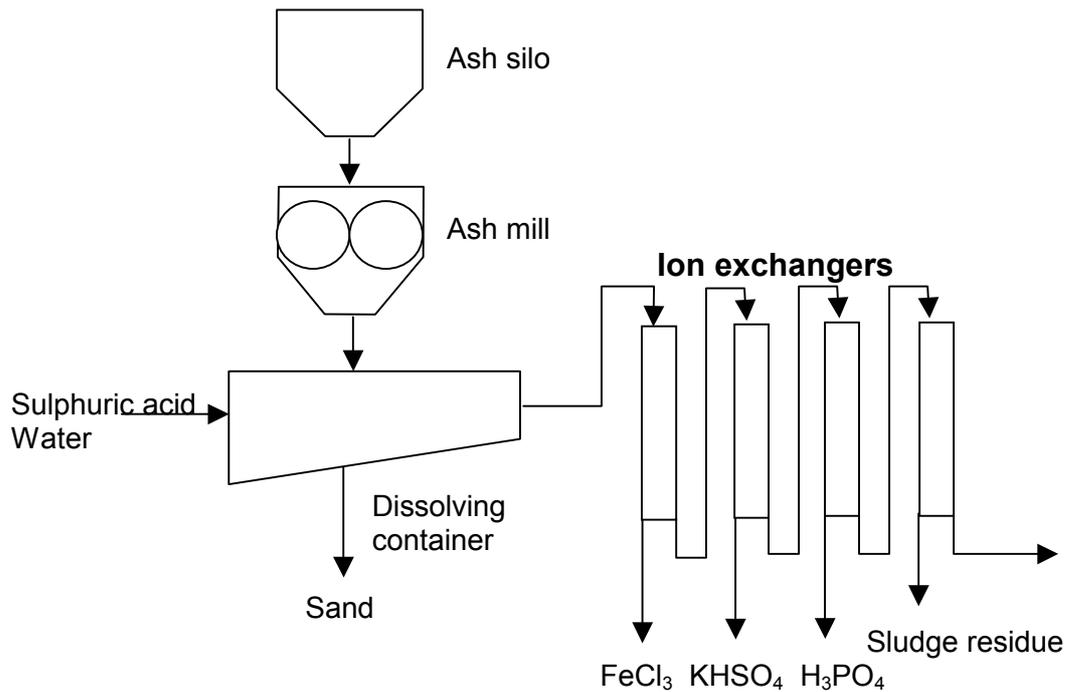
Figure 1. Resources recovery from ash with BioCon system (Svensson, 2000).

The ion exchange process shown by figure 2, is performed in four columns. In the first column, which is a cation exchanger is the ferric ions taken up. This column is regenerated with sulphuric acid producing ferric sulphate $Fe_2(SO_4)_3$. The second column is an anion exchanger, which removes sulphate ion, and is regenerated with potassium chloride producing potassium bisulphate, $KHSO_4$. The third column is an anion exchanger, which removes phosphate ions, and is regenerated with hydrochloric acid producing phosphoric acid, H_3PO_4. The last column is a cation exchanger, which removes other metals, and is regenerated hydrochloric acid producing metal chloride.
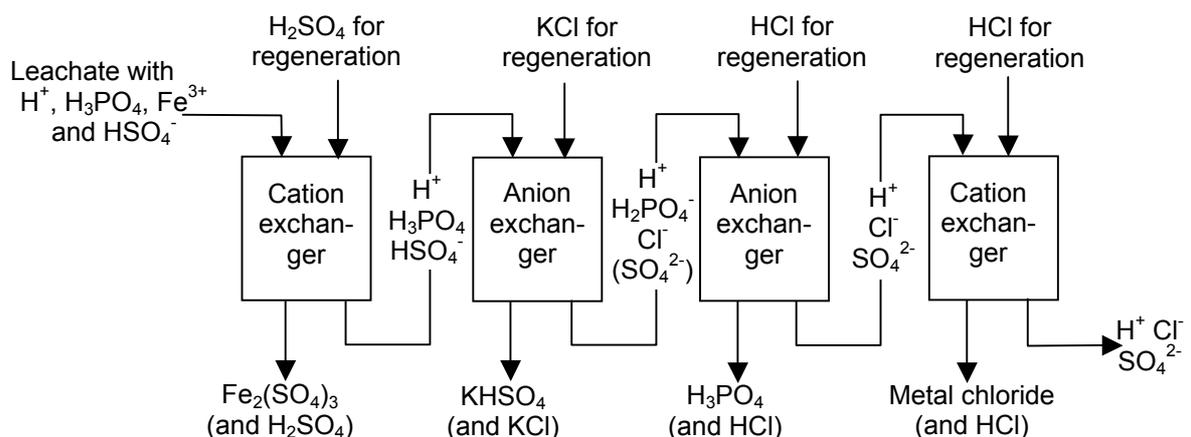

Figure 2. Regeneration of ion exchange columns for recovery of phosphoric acid.

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At pH-levels below 2 is phosphate present as zerovalent H_3PO_4, which not can be removed by ion exchange. The pH-level in the third column must therefore be above 2 there phosphate is converted to anions, $H_2PO_4^-$, which can be removed by an anion exchange column. At pH-levels above 2 is the potassium sulphate produced as K_2SO_4. The pH-level before the second column is below 2 and by removing the hydrogen sulphate ion the pH-level increases until the solution reach a pH-level there the phosphate is converted to monovalent ions which can be removed in the third column.

However, regeneration in one step often requires an excess amount of regenerate (Helfferich, 1995). The effluent regenerate from the column may contain a mixture of the product realised from the column and regenerate. The realised product must be separated from the regenerate by a further precipitation step and the remaining regenerate can be reused for regeneration. Alternatively can the need of excess amount of regenerate be reduced by reducing the degree of recovery. In the case of BioCon the degree of phosphorus recovery is about 60 % (Balmér et al, 2002). Using an anion exchange resin with a selectivity for chloride in the third phosphoric acid recovery column, almost all chloride ions in the regenerate will be exchanged for phosphoric acid at regeneration and an almost chloride free phosphoric acid will be produced. However, since the resin is selective for chloride only a part of the phosphoric acid in the leachate is taken up by the column, which reduces the degree of phosphorus recovery.

RemNut
Figure 2 shows the Rem-Nut process there phosphorus is recovered with ion exchange technology as struvite, magnesium ammonium phosphate ($MgNH_4PO_4$), from the effluent of a sewage treatment plant (Liberti et al., 2001). In the first column, which is a of zeolite type is ammonium ions exchanged for sodium ions. In the next column, which is an anion exchanger is phosphate exchanged for chloride ions. Both columns are regenerated with sodium chloride. The regenerate from the cation exchanger (sodium chloride solution with ammonium) is mixed with the med regenerate from the anion exchanger (sodium chloride solution with phosphate). Magnesium chloride is added to the mixture which produces a precipitation of magnesium ammonium phosphate. To increase the pH-level and promote struvite precipitation is also sodium hydroxide added. In the effluent from a sewage treatment plant is the ammonium/phosphate-ratio larger than required for struvite precipitation. Also phosphate must be added if nitrogen reduction shall be maximized by treating all of the effluent from the treatment plant in the cation exchanger. This form of RemNut (N-controlled RemNut) is in operation at the treatment plant in West Bari, Italy (Liberti et al., 1986). To avoid the cost of adding phosphate, only a partition of the effluent can be treated cation exchanger and the rest of the flow is lead directly to the anion exchanger. The flow through the cation exchanger is regulated to achieve an ammonium/phosphate-ratio which is optimal for struvite precipitation (P-controlled RemNut). In this case only 1/10 of the flow passes through the cation exchanger, which thereby can be smaller than the anion exchanger.
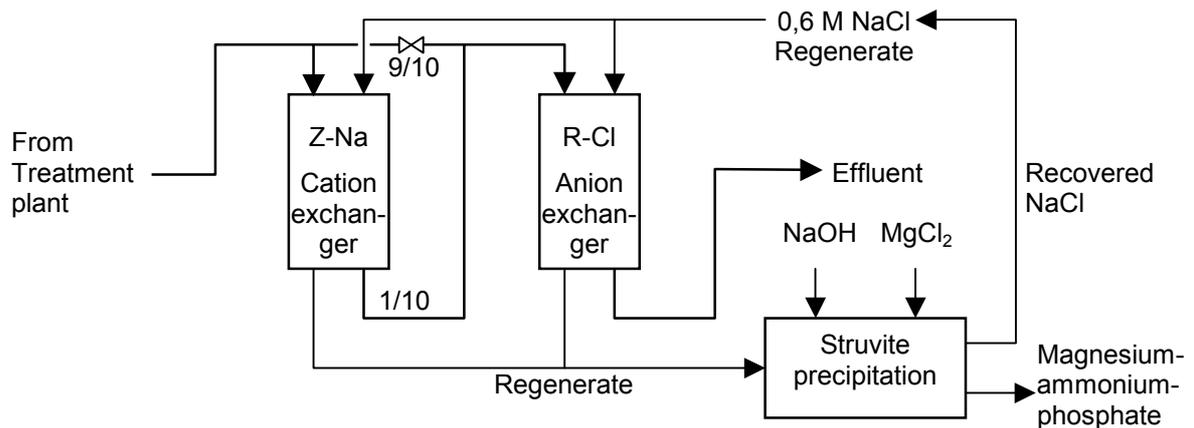
Figure 2. The Rem-Nut-process for recovery of phosphorus as struvite, magnesium ammonium phosphate (Liberti et al., 2001).

Aluminate ion exchange resins

Anion exchange resins of activated aluminium oxide can be used for recovering phosphate from a phosphate solution (Brattebø, 1983). At pH-levels below 8 - 8.5 aluminium oxide particles has a positive surface charge and can thus take up phosphate ions. Phosphate can be recovered by passing the solution containing phosphate through a column with aluminium oxide. To release the phosphate the pH-level is increased by addition sodium hydroxide. However, the solubility of the aluminium oxide increases with increasing pH-level. Therefore regeneration with a too strong sodium hydroxide solution gives loss of ion exchange resins. The concentration of the sodium hydroxide solution must thus adjusted to give a maximum phosphate release without a too large loss of ion exchange resins. The process has been tested in pilot plant scale (Donnert and Salecker, 1998). The aluminium oxide Al_2O_3 was activated at 600 °C and 0.5 molar sodium hydroxide was used for regeneration. The phosphorus was recovered by precipitation from the regenerate through addition of calcium hydroxide, and the remaining sodium hydroxide was reused for regeneration.

ACID LEACHING FOLLOWED BY SEPARATION WITH ION EXCHANGE

Separation with ion exchange columns

To get efficient phosphate recovery a low pH-value is needed to dissolve all metal phosphates. The dissolved phosphate is either zerovalent trihydrogen phosphate (phosphoric acid) or monovalent dihydrogen phosphate. If hydrogen chloride is used to dissolve ferric phosphate the solution will contain zero-valent phosphate together with ferric and chloride ions.

$$FePO_4 \text{ (s)} + 3\,H^+ + 3\,Cl^- \longrightarrow H_3PO_4^{\,o} + Fe^{3+} + 3\,Cl^-$$
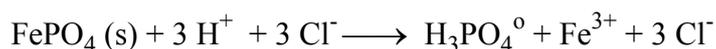
Ion exchange can be used to separate chloride and ferric ions from the solution. An ion exchanger (Helfferich, 1995) consists of solid material to which anions or cations in the solution can be adsorbed. The ion exchanger can be made of a polymer with attached ionic groups. Sulfonic, phosphonic and carboxylic groups are examples of ionic groups for cation exchangers. Amino groups were first used for anion exchangers. An cation exchanger have a material with a negative surface charge to which the metal cations in the solution (for instance iron ions) can be attached.

The zerovalent phosphate has a too low valency to be effectively removed by ion exchange. However, the ferric and other metal ions can as in the BioCon-process (Svensson, 2000) be separated with a cation exchanger.

$$CEX=H_3 + Fe^{3+} \longrightarrow CEX=Fe + 3\ H^+$$

And the chloride ions can be separated with an anion exchanger.

$$AEX=OH + Cl^- \longrightarrow AEX=Cl + OH^-$$

After separation of metal and chloride the phosphate will remain in the solution as phosphoric acid. Figure 3 shows a process scheme for leaching and separation with ion exchangers of metals from sludge or ash containing metal phosphate. After the leaching step the leachate first passes the cation exchanger before it goes to the anion exchanger. The metal ions must first be separated with a cation exchanger, before the chloride ions are taken up with an anion exchanger. Otherwise, the hydroxide ions released by the anion exchanger will raise the pH-level and metal phosphate will be precipitated in the anion exchanger column.

The minimum amount of chemicals needed for processing one mole of ferric phosphate, is three mole hydrochloric acid for leaching, three mole hydrochloric acid for regeneration of the cation exchanger and three mole sodium hydroxide for regeneration of the anion exchanger. 1 mole $FePO_4$, 6 mole HCl and 3 mole NaOH gives 1 mole H_3PO_4, 1 mole $FeCl_3$ and 3 mole NaCl. In the leaching an excess of acid is needed for decreasing the pH-level to the acidity needed for dissolving the metal phosphate.
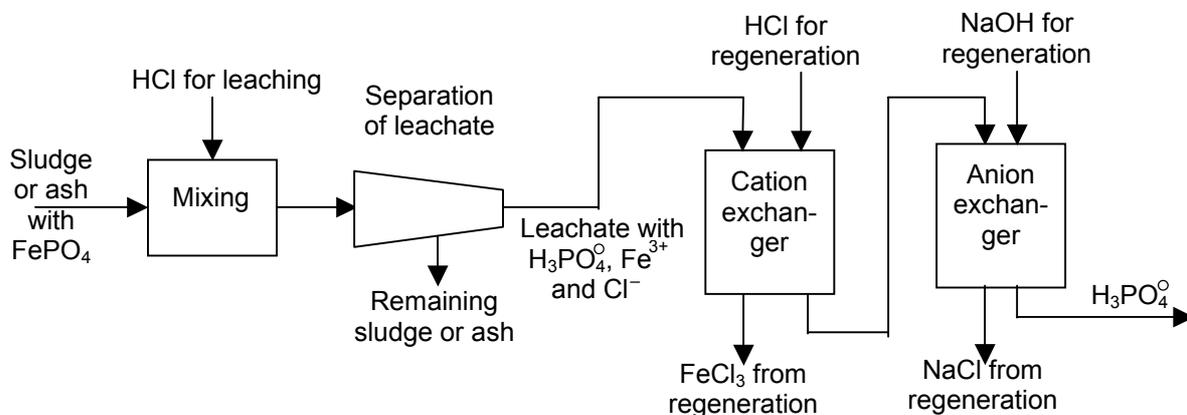

Figure 3. Process scheme for leaching and separation of metals from phosphate with ion exchangers.

The amount of chemicals needed for regeneration depends on how the regeneration is made. Figure 4 shows how counter flow of leachate, regeneration acid and ion exchange material can decrease the amount of acid needed for regeneration. For iron uptake the quota Fe^{3+}/H^+ must be higher in the leachate than in the ion exchange material and for regeneration the quota must be higher in the ion exchange material than in the regeneration acid.
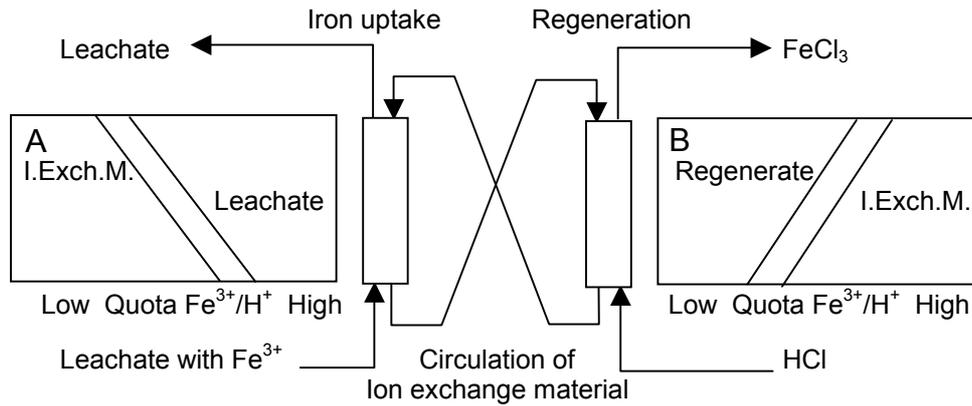
Figure 4. Counter flow of leachate, regeneration acid and ion exchange material decreases the amount of regeneration acid needed for regeneration. The diagrams A and B shows the quota Fe^{3+}/H^+ for leachate and ion exchange material during iron up take and regeneration.

If the leaching occurs at a pH-level above 2.2 there the phosphate is in form of monovalent dihydrogen phosphate, it can be removed with an anion exchanger.

$$AEX{=}OH + H_2PO_4^- \longrightarrow AEX{=}\,H_2PO_4 \;+\; OH^-$$
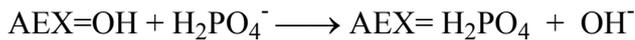
However since the anion exchanger preferentially will take up ions with higher valency, the solution must be free from other anions before only monovalent dihydrogen phosphate will be adsorbed. If an leaching acid with di- or trivalent anions is used, these anions can be removed in one anion exchanger and the monovalent dihydrogen phosphate in another anion exchanger. However, the leaching is most efficient at low pH-levels around 1 to 2 where the phosphate is zerovalent.

Raising the pH-level makes the phosphate mono or divalent, but also decreases the solubility for the phosphate. The anion exchanger can be substituted by phosphate precipitation with metal hydroxide. If calcium hydroxide is used, phosphate will be precipitated as calcium phosphate, which is the reverse reaction of producing phosphoric acid from apatite, used by the phosphate industry for phosphate fertilizer production.

$$2\,H_3PO_4^{\,o} + 3\,Ca^{2+} \longrightarrow Ca_3(PO_4)_2 + 6\,H^+$$

The minimum amount of chemicals needed for processing one mole of ferric phosphate, is three mole hydrochloric acid for leaching, three mole hydrochloric acid for regeneration of the cation exchanger and 1.5 mole calcium hydroxide for precipitation of phosphate. After precipitation the solution will contain hydrochloric acid, which may be reused for leaching phosphate or regenerating the cation exchanger, thus reducing the needed amount of hydrochloric acid to three moles. 1 mole $FePO_4$, 3 mole HCl and 1.5 mole $Ca(OH)_2$ give 0.5 mole $Ca_3(PO_4)_2$ and 1 mole $FeCl_3$.

Separation with ion selective membrane
Since phosphate is zero-valent phosphoric acid below pH-level 2, an electrochemical process with ion selective membrane may also be possible for separation of metals from a solution with phosphate dissolved by acid. The ion selective membrane is a membrane made of ion

exchange material. If anion exchange material is used an anion selective membrane is obtained and with cation exchange material a cation selective membrane is obtained. Ions can pass through the membrane by being adsorbed on one side and dissolved on the other. Cations, which are not adsorbed on anion exchange material, can thus not pass through an anion selective membrane and anions can not pass through a cation selective membrane. Since the ions are adsorbed on one side and dissolved on the other, the ion exchange material in the membrane must not be regenerated.

The driving force for ion transport through the membrane can be the difference in concentration. The ions are transported from a solution with high concentration to a solution with low concentration. A method for extracting copper and cadmium cation from phosphoric acid with cation selective membrane technique uses polyvinylsulfonic acid, which forms complexes with copper and cadmium (Eyal and Kislik, 1999, Kislik and Eyal, 2000). The polyvinylsulfonic is separated from the phosphoric acid by a cation exchange membrane. Due to the complex formation the copper and cadmium concentrations are low in polyvinylsulfonic acid and the cations are transported to the polyvinylsulfonic acid. In the next step copper and cadmium has to be removed from the polyvinylsulfonic acid. However, this process will not be suitable for separation of heavy metal from solutions with low metal concentrations.

An electric current can also be used as the driving force for ion transport through the membrane. Figure 5 shows electrochemical separation of copper with ion selective membrane (Juang and Lin, 2000). Complexing agents such as EDTA (ethylene-diamine-tetraacetic acid), NTA (nitrilotriacetic acid), citrate, and tartarate can be used to wash heavy metals. Heavy metals can be separated from the acid by chemical precipitation as hydroxide or sulphide. Due to creation of complexes the need of chemicals is large. Separation with a electrochemical membrane process can therefore be more convenient. Two electrodes are needed for the electrolyse, and copper is precipitated on the cathode electrode.
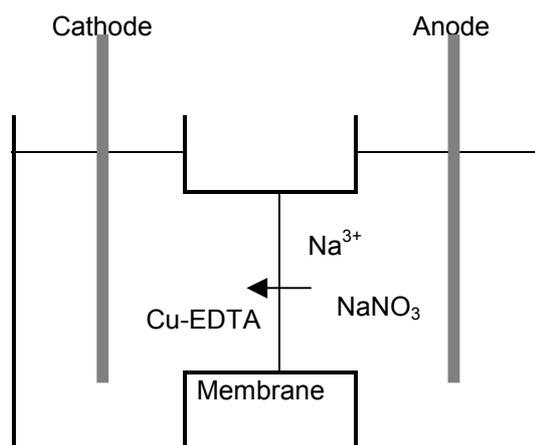
Figure 5. Electrochemical separation of copper with cation selective membrane.

$$Cu^{2+} + 2\ e^- \rightarrow Cu$$

To avoid oxidation of EDTA at the anode during electrolysis, the anode is separated from EDTA in a separate anode compartment, filled with sodium nitrate. At the electrode oxygen develops through from disintegration of water.

$$2H_2O\ \rightarrow\ O_2 + 4\ H^+ + 4\ e^-$$
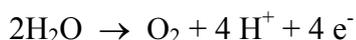
The anode compartment is separated from the cathode cell with EDTA by a cation selective membrane. The sodium ions are transported through the membrane and replace the copper ions, which are deposited on the cathode. Electrochemical separation through deposition on a cathode electrode can be used for precious metals as copper. The electrochemical separation

process should be useful for treatment of sludge with too high copper contents. The metals are leached from the sludge by complex forming acid, which is regenerated through the electrochemical separation process. If the process is to be used for treatment of sludge with non-precious metals, hydrogen evolution will occur at the cathode instead of metal deposition.

$$2H_2O + 2\,e^- \rightarrow H_2 + 2\,OH^-$$

To purify the complexing acid from non-precious metals the cathode cell must also be separated from the complexing acid by a cation selective membrane. The metal ions can pass through the membrane to the cathode cell. The metals will be precipitated in this cell as metal hydroxide together with the hydroxide ions produced in the cathode department.

COMBINED LEACHING AND SEPARATION WITH ION EXCHANGE

Magnetic ion exchange resins
Magnetic ion exchangers can be used for treatment of fluids with suspended solids, such as sludge or sewage water. If the fluid contains suspended solids an ion exchange column will be clogged with the solid particles. However, ion exchange resins with magnetic properties can be separated with magnets from the suspended solids. A process there magnetic ion exchange resin is mixed with sludge and separated with a magnetic drum has been studied by Swinton et al. (1989). Strong acid (sulphonic) exchangers were best for leaching metals from sludge. Weak acid (carboxyl) exchangers have no capacity below pH-level 4, and cannot be used. Chelating resins are more expensive. The process was used on sludge, but it should also be applicable on ash from incinerated sludge.

Removal of Pb, Cu, and Cd ions from diluted solutions by sorption onto clinoptilolite coprecipitated with magnetite, was studied by Feng et al. (2000). Using magnetic resins in a fluidized bed and separation through magnetic filtration, high levels of suspended solids can be tolerated. Ultrafine magnetite particles were used as magnetic seeding, and $FeCl_3$ was used as coagulant. The fine magnetite particles adhered to the clinoptilolite particle surfaces and could be removed by magnetic filtration. At a dosage of 0.5 gr/L clinoptilolite, 0.5–1.0 g of magnetite could render all clinoptilolite particles strongly magnetic. In this process ion exchange material is produced in the process and thus regeneration of the ion exchange material was not studied.

Use of the magnetic ion exchange process for recovery of phosphate and separation of metals from sludge or ash, reduces the chemical need significantly. Figure 6 shows a scheme for a process with leaching and separation of metals from phosphate with magnetic ion exchange resin. In mixing sludge with the magnetic cation exchange resin, hydrogen ions from the cation exchange material will dissolve the metal phosphate. The ion exchange material will take up the dissolved metal ions and phosphoric acid will remain in the solution.

$$CEX{=}H_3 + FePO_4\,(s) \longrightarrow CEX{=}Fe + H_3PO_4^{\ o}$$

The process is made in four steps.

1. Mixing ion exchange materials and sludge/ash.
2. Separation of phosphoric acid.
3. Separation of ion exchange materials.
4. Regeneration of the ion exchange materials.
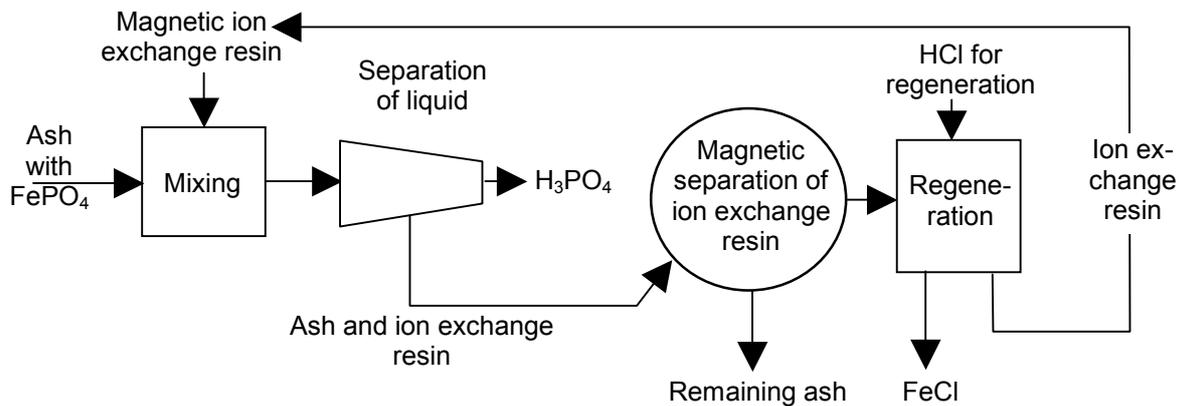
Figure 6. Process scheme for leaching and separation of metals from phosphate with magnetic ion exchange resin.

In this case there will be no need for an anion exchanger for removal of the anions from the leachate. The zerovalent phosphate will be in the leachate and can be separated from the sludge either before or after separation of ion exchange materials. The minimum amount of chemicals needed for processing one mole of ferrous phosphate, is three mole HCl for regeneration of the cation exchange resin. 1 mole $FePO_4$ and 3 mole HCl give 1 mole $H_3PO_4^o$ and 1 mole $FeCl_3$. Metal can be dissolved at higher pH-level since the ion exchange resin takes up dissolved metal ion and thus keeps the concentration of dissolved metal ions at a low level. This makes the need of acid for regeneration of the magnetic ion exchange resin smaller than the amount of acid needed for leaching.

Ion exchange textiles
Ion exchange textiles can be used for removal of heavy-metal from sludge (Sengupta and SenGupta, 1997). Figure 7 shows a continuous decontamination process where textile with cation exchange material is transported between two baths as a conveyer belt. In the first bath the textile takes up cations, which are realised in the second bath which contains regeneration acid. The ion exchange textiles textile can be used for phosphorus recovery in the same way as magnetic ion exchange resins. Hydrogen ions from the textile dissolves metals and phosphate from the sludge. The metals are taken up by the textile, thus leaving the phosphate dissolved in the solution. The textile can also be regenerated by use of an electrochemical cell (Basta et al., 1998). In the cell shown by figure 8, the textile passes between two cation selective membrane. An electric current is passed through the cell. Metal ions taken up by the textile goes through one membrane to the cathode and is replaced by hydrogen ions coming in through the other membrane from the anode.
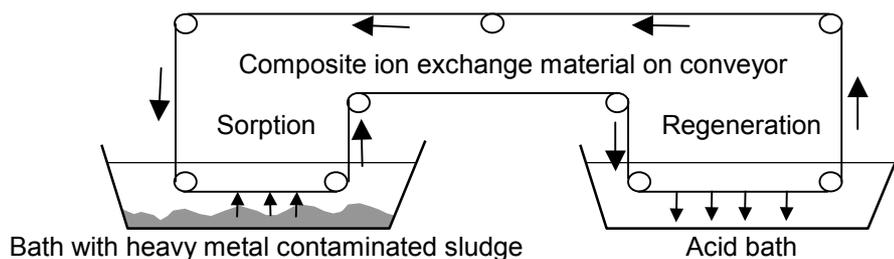
Figure 7. A continuous decontamination process for heavy-metal removal from a sludge reactor (Sengupta and SenGupta, 1997).
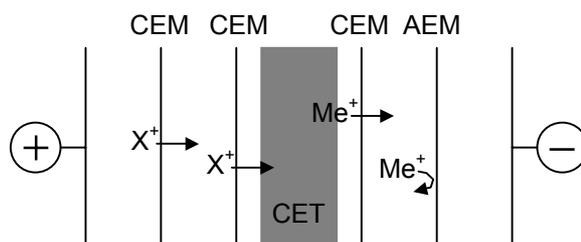

Figure 8. Regeneration of cation exchange textile with an electrolytic cell (Basta et al., 1998). CET is cation exchange textile, CEM is cation selective membrane and AEM is anion selective membrane.

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