Title: Can limestone, steel slags or man-made sorption materials be used to enhance phosphate-phosphorus retention in treatment wetland for peat extraction runoff with low phosphorous concentration?

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Abstract

This study examined possibilities to enhance phosphorus (P) retention in wetlands using different materials that could enhance removal of phosphate P (PO\textsubscript{4}-P) from runoff waters with fairly low P concentrations (P\textsubscript{tot} average 80-90 μg L\textsuperscript{-1} and PO\textsubscript{4}-P 25-30 μg L\textsuperscript{-1}) typical for peat extraction runoff. The retention potential of sorption materials, that had previously shown good retention capacity was first studied in laboratory batch tests using steel slag (basic oxygen furnace slag (BOF)), Filtralite\textsuperscript{®}P (high Ca and Mg clay), CFH 12 (ferrihydroxide), limestone, Phoslock\textsuperscript{®} (95% bentonite clay material + 5% lanthanum) and iron gypsum in year 2010. Based on batch test results and material properties (column tests not suitable for fine clay materials such as Phoslock\textsuperscript{®}), steel slag, CFH 12 and iron gypsum products were selected for column tests. The columns experiments were run for almost three months during spring 2011. Steel slag and Phoslock\textsuperscript{®} were selected for
further testing \textit{in situ} in a treatment wetland. In the laboratory set-ups, all materials tested  
retained PO$_4$-P (70-90\% in batch tests and approximately 10-80\% in column experiments). However, in the field scale set-up, neither steel slag nor Phoslock®  
successfully retained PO$_4$-P. The reasons may be e.g. for steel slag, too low pH, too large  
grain size, and too short retention time. Also, for some set-up, the given instruction were  
not followed during construction works. Further studies are needed to test different  
particle sizes and new potential materials for retaining P in treatment wetlands with high  
hydraulic loading rate, low P concentration and low pH.

\textbf{Keywords}

Phosphorus, Sorption material, Treatment wetland, Peat, Retention

1. Introduction

Treatment wetlands (TWs) are globally used for wastewater purification purposes, and  
their ability to retain suspended solids (SS) is usually high (70-90\%), but the ability to  
retain phosphorus (P) is lower (30-60\%) (Vymazal, 2010). In Finland, TWs have mainly  
been constructed on intact peatlands, where they can retain on average 40-55\% of  
phosphorus (P) from peat extraction runoff (Heikkinen et al., 2002; Kløve et al., 2012).  
In addition, TWs are used for polishing municipal wastewater, and TWs constructed on  
pristine peatlands show P retention of 12-83\% when the hydraulic load range from of 21-  
44 mm d$^{-1}$ (Ronkanen and Kløve, 2009). According to the recent Finnish national peatland  
use strategy, peat extraction areas should be established on previously drained peatlands  
with little ecological value. Thus treatment wetlands are also often constructed on drained  
peatlands. According to Postila et al. (2014), some of the TWs constructed on drained
areas retain phosphate phosphorus (PO$_4$-P) nearly as well as wetlands constructed on intact areas. However, in the same study some of the TWs released PO$_4$-P, especially during the first years after their establishment. Phosphorus release after rewetting of drained peatlands in wetland restoration has also been observed in other studies (Kieckbusch and Schrautzer, 2007; Koskinen et al., 2011; Nieminen et al., 2005). P leaching may partly occur due to reduction of iron (Fe) under anaerobic conditions, resulting in PO$_4^{3-}$ releases (Reddy and DeLaune, 2008). Also high P concentration in surface peat layer (Postila et al., 2014) and the death of forest species (Jauhiainen et al., 2002) can lead to P leaching after rewetting.

Possibilities to enhance P retention in TWs have been studied with different materials mainly at laboratory scale (Vohla et al., 2011). The factors found to affect P retention in sorption materials are e.g. material grain size (e.g. Xu et al., 2006), P inflow concentration (e.g. Lyngsie et al., 2015), pH (e.g. Oliveira et al., 2015), hydraulic loading rate (Vohla et al., 2011), retention time (e.g. Lyngsie et al., 2015; Penn et al., 2012), water velocity (Claveau-Mallett et al., 2012), and organic matter content (e.g. Xu et al., 2006). Sorption materials can be divided into: 1) natural materials, such as the siliceous sedimentary rock opoka (Johansson and Gustafsson, 2000), shell sand (Ådåm et al., 2007) and peat (e.g. Kõiv et al., 2009); 2) industrial by-products, such as slags (e.g. Johansson 1999; Vohla et al., 2011) and ash (e.g. Xu et al., 2006); and 3) man-made products, such as Filtralite P® (Ådåm et al., 2005; Vohla et al. 2011). The materials can also be divided according to their chemical composition into: a) materials which contain metals (such as aluminium (Al) and Fe); b) materials which contain soluble divalent earth metals (calcium (Ca) and magnesium (Mg)); and c) mixtures of these (Klimeski et al., 2012). By-product materials
such as slags have been frequently tested, because they are widely available and cheap materials. However, based on Klimeski et al. (2012) review, P sorption tests are usually conducted using water with a high P concentration, whereas the average P concentrations in peat extraction runoff are 80-90 μg P_{tot} L^{-1} and 25-30 μg PO_4-P L^{-1} (Postila et al., 2014; Tuukkanen et al., 2012).

Addition of Fe, Al and Ca can increase PO_4-P retention due to their sorption capacity (Richardson, 1985; Seo et al., 2005). However, Fe and Al sorb P in acidic conditions, whereas Ca sorbs P under near-neutral conditions (Reddy and DeLaune, 2008). The aim of this study was to determine whether addition of sorption materials can enhance PO_4-P retention in TWs constructed for purification of peat extraction runoff. Batch and column tests were first carried out in the laboratory and then promising candidate materials were tested in situ in a TW. Our starting hypothesis was that sorption materials can retain P in laboratory and field conditions.

2. Material and methods

2.1 Laboratory tests

Different sorption materials for retaining PO_4-P were first studied in the laboratory in batch (year 2010) and column (year 2011) tests. The materials studied in batch tests were selected based on a literature review considering material cost-effectiveness, retention efficiency, particle size, availability and costs. Locally available materials and by products were carefully considered to avoid e.g. high transport costs and steel slag (basic oxygen furnace slag (BOF) including mainly calcium oxide (CaO), Fe and silicon dioxide (SiO_2)) from Raahe Rautaruukki Oyj (nowadays SSAB) was chosen. Filtralite®P (a clay
material with high Ca and Mg content), CFH 12 (ferrihydroxide), limestone (mainly \( \text{CaCO}_3 \)), Phoslock\textsuperscript{®} (95% bentonite clay material + 5% lanthanum), and iron gypsum from Kemira Oyj was also selected as promising materials. In these batch tests, runoff from a peat extraction area (8 µg PO\textsubscript{4}-P L\textsuperscript{-1}) and pore water from the peat or mineral layer of TWs (40 and 250 µg PO\textsubscript{4}-P L\textsuperscript{-1}) were used. The pH in these waters varied from 5.6 to 6.4, with the highest values occurring in runoff water. The batch test on each material was performed with 100 g of sorption material and 200 mL of water in four parallel samples (replicates), because ratio represent more realistically the field conditions. We followed the protocol described by Heikkinen et al. (1995): the mixture of sorption material and water was first shaken mechanically for one hour (shaker speed was selected between 300-400 min\textsuperscript{-1} in order to have all the tested mass be in circulation in the bottle), followed by a 23-hour standing time. After this, the samples were shaken for 10 minutes and centrifuged for 15 minutes at 4000 revolutions per minute. Finally, the samples were filtered through a Whatman\textsuperscript{®} GF/C filter (pore size 1.2 µm) and a Gelman filter (pore size 0.45 µm). The samples were analysed for e.g. PO\textsubscript{4}-P, pH, Al, Fe, Ca, Mg and sulphur (S) in an accredited laboratory.

Column tests were run with steel slag (grain size 3-10 mm and 10-20 mm, which were used in earlier studies also), CFH 12 and Sachtofer PR (iron gypsum material from Sachtleben Pigments Oy) with peat extraction runoff water with an above-normal PO\textsubscript{4}-P concentration (650 µg PO\textsubscript{4}-P L\textsuperscript{-1}). In the tests, two different-sized columns were used, so that the diameter was at least 5 time higher than particle size (CEN ISO/TS 17892-11:fi), but the needed water amount was minimized during test. In this way the difficulties in collecting water and storing it were minimized. Columns with inner diameter 10.4 cm and height 20.8 cm were used for steel slag with grain size 10-20 mm and Sachtofer PR, due
to their larger particle sizes, while columns with inner diameter 5.2 cm and height 31 cm were used for steel slag with grain size 3-10 mm and for CFH 12. The height of these sorption materials in the columns was about 15 cm. A filter paper (Whatman 114) was installed in smaller columns and a filter mesh (pore size 0.5 mm) in larger columns to prevent sorption material from ending up in the outlet pipe. In smaller columns, a quartz sand layer (grain size 3-5 mm) was also added below (thickness 3 cm) and above (1.5 cm) the sorption material to ensure uniform water flow. Because the grain size of quartz sand was <10-20 mm, this material was not used in larger columns. One column per sorption material was used and the water was fed continuously by pump from the bottom of these columns from 1 m³ containers that were continuously mixed by a small pump. The target outflow for small columns was 2 mL min⁻¹ and for large columns 8 mL min⁻¹. Thus the mean water residence time was calculated to be approximately similar (one hour) in all columns. This residence time was used as it was possible to achieve also in field conditions where the water discharge is high. However, the actual mean residence time in columns varied from 1 hour to 1.5 hours and the flow velocity varied from 1.6 to 1.9 mL min⁻¹ in the small columns and from 5 to 8 mL min⁻¹ in the bigger columns. When the flow velocity was noticed to be clearly under the target outflow (2 or 8 mL min⁻¹), the pumping speed was increased. These experiments were done in hydraulically saturated conditions and run for almost three months (83 days).

The hydraulic conductivity (K) during the column testes was checked regularly to determine clogging effects. First the K was first determined using empty columns where was only filter mesh (large column) or filter paper and a quartz sand layer (small column). The aim of that was to check how the column system restricted water flow (affected
hydraulic conductivity). After that, the hydraulic conductivity was determined for the filled columns first one day after the beginning of the test, then approximately every week for two first months, and finally at the end of the test. The hydraulic conductivity (K-value) was calculated as (Tie- ja vesirakennushallitus, 1974):

\[ K = \frac{Q \cdot L}{t \cdot A \cdot H} \]  (1)

where Q is the amount of water (cm³) flowing through the column sample at certain time t (s), L is the height of sample (cm), A is the surface area of sample (cm²) and H is the pressure height (cm). Q was measured in the space without sorption materials on the top of the column. On the occasions when hydraulic conductivity was measured i.e. approximately every week for two first months, and finally at the end of the test, inflow and outflow samples were also taken and analysed for PO₄-P, total P, pH, Ca, Al, Fe, S, Mg, manganese (Mn), potassium (K), sodium (Na), barium (Ba), strontium (Sr), titanium (Ti) and zinc (Zn) in an accredited laboratory. At the beginning of the test, samples were taken from the water containers supplying inflow water for analyses of total nitrogen (N) (2300 µg L⁻¹), total P (670 µg L⁻¹), chemical oxygen demand (CODMn) (14 mg L⁻¹), suspended solids (SS) (26 mg L⁻¹) and pH (6.5).

2.2. In situ tests

Steel slag and Phoslock® were also tested in situ in Äijönneva TW (64°5’13”N 25°4’56”E) in northern Finland. Steel slag was selected because it is a cheap by-product, available near the study site, can retain PO₄-P based on laboratory tests and does not leach sulphur. Steel slag filters were constructed in the collection ditch as oxic drains to enhance P
retention. Slag was also placed in small heaps in front of perforated pipe holes, by request of the peat extraction company who wanted to test this alternative. Phoslock® was selected because it is known to retain PO₄-P well and has previously been used in lake restoration projects. In lake restoration it is spread on the bottom of lake, where it binds PO₄-P from the water column and the P released from the sediment. Phoslock® was spread on top of the wet and partially water covered peat surface.

The Äijönneva area was drained some decades ago and is now covered with a dense tree stand. The runoff water from the peat extraction area (surface area 103 ha) is pumped through perforated pipes to the TW from June 2009 onwards. The wetland area (5.8 ha) consists of two parts (southern and northern) separated by a main ditch called Äijönkanava (Fig. 1). The average PO₄-P inflow concentrations were 20 µgL⁻¹, 50 µgL⁻¹ and 25 µgL⁻¹ in summer 2009, 2010 and 2011, respectively. PO₄-P leaching from the TW was observed during summer in these years, raising concerns as the environmental permit expects P retention. The average daily outflow discharge from the TW, based on Finnish Environment Institute hydrological watershed model system (WSFS; Finnish Environment Institute, 2013), was 412 L d⁻¹ in summer and 1220 L d⁻¹ in autumn 2011. The average inflow pH was 6.6, 6.6 and 6.5 and the average outflow pH was 6.0, 5.0 and 6.2 in summer 2009, 2010 and 2011, respectively. Other information on water quality and purification efficiency results for Äijönneva TW can be found in Postila et al. (2014).
Fig. 1. Sketch of the Äijönneva wetland showing the position of perforated pipes and collector ditch, the locations of the two sorption materials tested (steel slag and Phoslock®) and the locations of water sampling points. The runoff water flows to the wetland via perforated pipes. On the wetland, the water partly flows in the surface peat layer or on soil surface, and partly via ditches. On the northern part of Äijönkanava channel, water flows to the collector ditch and finally to the outflow discharge point. On
the southern site of Äijönkanava channel, the water flows to the channel and finally to the outflow point.

In late August-early September 2011, three steel slag oxic drains were constructed in the collector ditch in the northern part of the wetland (Fig. 1). These oxic drains were 6-7 m long and filled with 140 t steel slag with the grain size varying from 10 to 80 mm. The grain size of steel slag used in oxic drains was larger than used in the laboratory test (varying from 10 to 20 mm) as it was not possible to obtain large amounts of the finer-grained steel slag used in the laboratory trial. Also ten tonnes of steel slag with grain size 7-15 mm were spread as small heaps in front of one perforated pipe outlet holes in the northern part of the wetland because it was the other grain size, which was possible to get field-scale amounts, and not so big than 10-80 mm. In addition, one tonne of Phoslock® was spread around another perforated pipe.

Water quality was monitored in ditches below each test area and water flow outlets from the control area (Fig. 1). The southern part of the wetland was then used as a control area, because the water quality was quite similar to that in the northern part in autumn 2010. In autumn 2011, water from all study points was analysed bi-weekly for pH, PO₄-P, total P and dissolved total P in an accredited laboratory (Fig. 1). Wetland outflow water was analysed once per month for Fe, Al, Mn, vanadium (V), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb) and nickel (Ni), in order to monitor possible metal leaching from the steel slag material. The water residence time in steel slag oxic drains was determined using sodium chloride (8 kg NaCl in 150 L water) as tracer and measuring the changes in electric conductivity in water.
3. Results and discussion

3.1 Batch tests

Based on the batch test results, all sorption materials studied can retain PO₄-P at rates varying from 70% to 100% for the 24 h contact time used (Table 1). The purification efficiency was lowest (70-93%) for limestone, while for the other materials it ranged from 88% up to 100%. During the batch test, the water became alkaline when steel slag or Filtralite®P was used and it became acidic when CHF 12 was used. With the other materials, the pH stayed neutral. However, CFH 12 and iron gypsum leached noticeable amounts of S (Table 1).

Table 1. PO₄-P retention (%) and mean values of water quality parameters measured after batch tests. Minimum and maximum values are given in brackets.

<table>
<thead>
<tr>
<th>Sorption material</th>
<th>PO₄-P retention efficiency (%)</th>
<th>pH</th>
<th>S (mg L⁻¹)</th>
<th>Mg (mg L⁻¹)</th>
<th>Fe (µg L⁻¹)</th>
<th>Ca (mg L⁻¹)</th>
<th>Al (µg L⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel slag</td>
<td>98-99 (12.1-12.2)</td>
<td>12.2</td>
<td>1.2 (&lt;0.1)</td>
<td>18 (5-23)</td>
<td>620 (580-660)</td>
<td>120 (98-150)</td>
<td></td>
</tr>
<tr>
<td>Filtralite®P</td>
<td>93-100 (12.2-12.3)</td>
<td>12.3</td>
<td>9.4 (&lt;0.1)</td>
<td>6 (5-13)</td>
<td>670 (660-690)</td>
<td>590 (530-640)</td>
<td></td>
</tr>
<tr>
<td>CFH 12</td>
<td>98-100 (4.4-5.5)</td>
<td>4.6</td>
<td>2700 (2600-2700)</td>
<td>1800 (1800-1900)</td>
<td>59 (41-87)</td>
<td>280 (260-300)</td>
<td>17 (13-21)</td>
</tr>
<tr>
<td>Limestone</td>
<td>70-93 (7.6-8.0)</td>
<td>7.9</td>
<td>0.81 (0.59-10)</td>
<td>3 (3-4)</td>
<td>2100 (1800-2500)</td>
<td>38 (28-50)</td>
<td>120 (29-210)</td>
</tr>
<tr>
<td>Phoslock®</td>
<td>88-99 (5.6-6.7)</td>
<td>6.4</td>
<td>16 (15-17)</td>
<td>20 (7-40)</td>
<td>&lt;5 (&lt;5-11)</td>
<td>170 (57-350)</td>
<td>2 (1-3)</td>
</tr>
<tr>
<td>Iron gypsum</td>
<td>98-99 (7.5-7.8)</td>
<td>7.7</td>
<td>610 (550-640)</td>
<td>63 (56-76)</td>
<td>17 (7-30)</td>
<td>530 (520-550)</td>
<td>30 (26-34)</td>
</tr>
</tbody>
</table>
3.2 Column tests

In the column tests, PO$_4$-P purification efficiency varied as a function of time (Fig. 2). As expected based e.g. on Johansson (1999) and Xu et al. (2006), higher retention capacity was observed for small particle-sized sorption materials (steel slag 3-10 mm and CFH 12). In addition, the Sachtofer PR iron gypsum material retained PO$_4$-P quite well except at the end of the test, when PO$_4$-P purification efficiency was 0% and also Al, Fe, Mn, Zn and Ti were leached. Typically, some decrease in the purification efficiency was observed with time, caused by the cumulative P input and saturation, which can be expected (e.g. Àdám et al., 2007; Shilton et al., 2006; Søvik and Kløve, 2005). The difference in outflow water pH between columns was greatest in the beginning of the test (Fig. 3). The pH of water increased after contact with the steel slag material, which contains soluble CaO that can elevate the water pH on dissolution (Klimeski et al., 2012). In the beginning of the test, Sachtofer PR leached more than 600 mg S L$^{-1}$ and CFH 12 over 200 mg S L$^{-1}$. The Sachtofer PR column also leached S (160 mg L$^{-1}$) at the end of test. The Ca content in water from the Sachtofer PR column was elevated during the whole test period. In the columns of the other materials tested it was also elevated, but only in the beginning of the tests. Total P, Al, Fe, Mn, Zn, Na, Ba, Sr, Mg, K and Ti concentrations in column outflow were typically under or at the inflow level except for some individual samples, but Mn, Sr and Mg concentrations in outflow from Sachtofer PR were generally elevated.

Hydraulic conductivity in the small particle size materials tested was lower than in the larger particle size materials (Fig. 4), as previously observed by Penn et al. (2012). The small particle size materials can also become clogged easily (Vohla et al., 2011). The hydraulic conductivity of CFH was low during the test period. However, in the steel slag
(3-10 mm) column the hydraulic conductivity suddenly increased noticeably. This was probably caused by development of a preferential flow path inside the column, and did not represent the real hydraulic conductivity in the material. During the test period there was some variation in hydraulic conductivity, which was probably partly caused by clogging and dissolution of clogged material. For all test runs, the K-values observed were smaller than for empty columns (including only filter mesh or filter paper and quartz sand), where the conductivity was 0.05 cm s\(^{-1}\) (larger columns) and 0.08 cm s\(^{-1}\) (smaller columns), so the equipment did not affect the sorption materials hydraulic conductivity.

![Fig. 2. PO\(_4\)-P retention (%) in different columns during the column tests.](image1)

![Fig. 3. pH values in the inflow and outflow of different columns during the column tests.](image2)
Fig. 4. Hydraulic conductivity (cm s\(^{-1}\)) in different columns during the column tests.

3.3 In situ tests in a treatment wetland

Based on water samples from the TW, the constructed sorption systems did not retain detectable amounts of PO\(_4\)-P (Fig. 5), total P or dissolved total P. In the ditch below the Phoslock\® area, the PO\(_4\)-P concentrations were sometimes smaller than in the other areas. Moreover, the Cd, Cr, Cu, Ni and Pb concentrations were under the detection limit in all outflow samples. The Mn and V concentrations were slightly elevated after the sorption materials were installed, but their concentrations returned to their initial level by the end of autumn at the latest. The Fe concentrations in outflow were lower than in inflow. Hence there was no significant metal leaching from the sorption materials.
Fig. 5. PO$_4$-P concentrations (µg L$^{-1}$) in wetland water before and after sorption materials were installed in Äijönneva wetland.

The residence time of water was only 10-20 minutes in steel slag oxic drains. This is probably too short for efficient PO$_4$-P retention. The low P reduction efficiency may also have been partly caused by large particle size (10-80 mm), as higher reactive surface area (i.e. smaller particle size) has been observed to enhance P retention (Johansson, 1999; Xu et al., 2006). The pH at different measurement points (Fig. 1) was mainly at least slightly acidic, except two samples for point 3 (8.9.2011 (pH 7.5) and 17.10.2011 (pH 7.1)) and point 5 samples, in which pH varied from 6.8 to 7.2 (21.9.-16.11.2011). On 8.9.2011, the outflow pH was also 7.0. The formation of Ca-phosphate precipitates is efficient in alkaline conditions (Johansson and Gustafsson 2000). Thus Ca-rich steel slag was not a suitable material for P retention in the acidic conditions which occur in Äijönneva TW. Klimeski et al. (2012) also noted that Ca-rich sorption materials can retain P well in the...
laboratory, but perhaps not in the field. When dissolved Ca\(^{2+}\) contact with atmospheric CO\(_2\) this can also lead formation of CaCO\(_3\) precipitates (Drizo et al., 2002).

The small heaps of steel slag in the front of the perforated pipe holes may not have worked for the same reason as the steel slag oxic drains. However, if these heaps are able to retain some PO\(_4\)-P from inflow, it is possible that more PO\(_4\)-P is leached from the wetland peat before the water flows to the wetland ditch. The Phoslock\(^{\circledR}\) material contains mostly clay, which can harden during a dry period following a wet (rainy) period. Therefore it should be spread as evenly as possible on the wetland surface. However, here it ended up being applied in small heaps near one of the perforated pipes, the given instruction were not fully understood and followed during construction works. Thus the Phoslock\(^{\circledR}\) formed much less reactive surface area for P retention than could have provided if it were spread evenly, as initially planned. As a result, this probably reduced the PO\(_4\)-P purification potential of this material.

4 Conclusions

Sorption materials containing P-precipitating metals can be used for purification of wastewater. We tested six different materials in laboratory experiments first in batch test, and then selected the most promising materials for column tests runs (as Phoslock\(^{\circledR}\) is a fine clay powder it was not used in column runs). Based on the laboratory test results and considering steel slag as a cost-efficient by-product, steel slag and Phoslock\(^{\circledR}\) was tested in field experiments. The results indicated that PO\(_4\)-P can be retained by sorption materials under laboratory conditions, although due to cumulative P input into sorption material columns, the sorption capacity can decrease over time. When steel slag and
Phoslock® were added to a treatment wetland, clear enhanced P purification was not observed. The reasons for this may be too large grain size of the steel slag material used, and consequently too short water retention time in this sorption material. In addition, the pH of water passing through the steel slag was too low for Ca-rich sorption material to precipitate P. It was concluded that sorption materials such as Phoslock®, which can be spread on the wetland surface, should be evenly distributed to gain the most efficient P sorption. Further studies are needed to identify suitable sorption materials and material particle sizes for acidic field conditions with high hydraulic loading rate and low P concentration. Overall, converting drained peatlands to efficiently working treatment wetlands remains a challenge for research and water protection actions.

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