1 **Title: Can limestone, steel slags or man-made sorption materials be used to enhance**

2 **phosphate-phosphorus retention in treatment wetland for peat extraction runoff**

3 **with low phosphorous concentration?**

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12

13 **Abstract**

14 This study examined possibilities to enhance phosphorus (P) retention in wetlands using

15 different materials that could enhance removal of phosphate P (PO_4-P) from runoff waters

16 with fairly low P concentrations (P_{tot} average 80-90 $\mu g\ L^{-1}$ and PO_4-P 25-30 $\mu g\ L^{-1}$) typical

17 for peat extraction runoff. The retention potential of sorption materials, that had

18 previously shown good retention capacity was first studied in laboratory batch tests using

19 steel slag (basic oxygen furnace slag (BOF)), Filtralite®P (high Ca and Mg clay), CFH 12

20 (ferrihydroxide), limestone, Phoslock® (95% bentonite clay material + 5% lanthanum)

21 and iron gypsum in year 2010. Based on batch test results and material properties (column

22 tests not suitable for fine clay materials such as Phoslock®), steel slag, CFH 12 and iron

23 gypsum products were selected for column tests. The columns experiments were run for

24 almost three months during spring 2011. Steel slag and Phoslock® were selected for

25   further testing *in situ* in a treatment wetland. In the laboratory set-ups, all materials tested

26   retained PO_4-P (70-90% in batch tests and approximately 10-80% in column

27   experiments). However, in the field scale set-up, neither steel slag nor Phoslock®

28   successfully retained PO_4-P. The reasons may be e.g. for steel slag, too low pH, too large

29   grain size, and too short retention time. Also, for some set-up, the given instruction were

30   not followed during construction works. Further studies are needed to test different

31   particle sizes and new potential materials for retaining P in treatment wetlands with high

32   hydraulic loading rate, low P concentration and low pH.

33

34   **Keywords**

35   Phosphorus, Sorption material, Treatment wetland, Peat, Retention

36

37   **1. Introduction**

38   Treatment wetlands (TWs) are globally used for wastewater purification purposes, and

39   their ability to retain suspended solids (SS) is usually high (70-90%), but the ability to

40   retain phosphorus (P) is lower (30-60%) (Vymazal, 2010). In Finland, TWs have mainly

41   been constructed on intact peatlands, where they can retain on average 40-55% of

42   phosphorus (P) from peat extraction runoff (Heikkinen et al., 2002; Kløve et al., 2012).

43   In addition, TWs are used for polishing municipal wastewater, and TWs constructed on

44   pristine peatlands show P retention of 12-83% when the hydraulic load range from of 21-

45   44 mm d^{-1} (Ronkanen and Kløve, 2009). According to the recent Finnish national peatland

46   use strategy, peat extraction areas should be established on previously drained peatlands

47   with little ecological value. Thus treatment wetlands are also often constructed on drained

48   peatlands. According to Postila et al. (2014), some of the TWs constructed on drained

49   areas retain phosphate phosphorus (PO_4-P) nearly as well as wetlands constructed on

50   intact areas. However, in the same study some of the TWs released PO_4-P, especially

51   during the first years after their establishment. Phosphorus release after rewetting of

52   drained peatlands in wetland restoration has also been observed in other studies

53   (Kieckbusch and Schrautzer, 2007; Koskinen et al., 2011; Nieminen et al., 2005). P

54   leaching may partly occur due to reduction of iron (Fe) under anaerobic conditions,

55   resulting in PO_4^{3-} releases (Reddy and DeLaune, 2008). Also high P concentration in

56   surface peat layer (Postila et al., 2014) and the death of forest species (Jauhiainen et al.,

57   2002) can lead to P leaching after rewetting.

58

59   Possibilities to enhance P retention in TWs have been studied with different materials

60   mainly at laboratory scale (Vohla et al., 2011). The factors found to affect P retention in

61   sorption materials are e.g. material grain size (e.g. Xu et al., 2006), P inflow concentration

62   (e.g. Lyngsie et al., 2015), pH (e.g. Oliveira et al., 2015), hydraulic loading rate (Vohla

63   et al., 2011), retention time (e.g. Lyngsie et al., 2015; Penn et al., 2012), water velocity

64   (Claveau-Mallet et al., 2012), and organic matter content (e.g. Xu et al., 2006). Sorption

65   materials can be divided into: 1) natural materials, such as the siliceous sedimentary rock

66   opoka (Johansson and Gustafsson, 2000), shell sand (Àdàm et al., 2007) and peat (e.g.

67   Kõiv et al., 2009); 2) industrial by-products, such as slags (e.g. Johansson 1999; Vohla et

68   al., 2011) and ash (e.g. Xu et al., 2006); and 3) man-made products, such as Filtralite P[®]

69   (Àdàm et al., 2005; Vohla et al. 2011). The materials can also be divided according to

70   their chemical composition into: a) materials which contain metals (such as aluminium

71   (Al) and Fe); b) materials which contain soluble divalent earth metals (calcium (Ca) and

72   magnesium (Mg)); and c) mixtures of these (Klimeski et al., 2012). By-product materials

73　such as slags have been frequently tested, because they are widely available and cheap

74　materials. However, based on Klimeski et al. (2012) review, P sorption tests are usually

75　conducted using water with a high P concentration, whereas the average P concentrations

76　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;

77　Tuukkanen et al., 2012).

78

79　Addition of Fe, Al and Ca can increase PO_4-P retention due to their sorption capacity

80　(Richardson, 1985; Seo et al., 2005). However, Fe and Al sorb P in acidic conditions,

81　whereas Ca sorbs P under near-neutral conditions (Reddy and DeLaune, 2008). The aim

82　of this study was to determine whether addition of sorption materials can enhance PO_4-P

83　retention in TWs constructed for purification of peat extraction runoff. Batch and column

84　tests were first carried out in the laboratory and then promising candidate materials were

85　tested *in situ* in a TW. Our starting hypothesis was that sorption materials can retain P in

86　laboratory and field conditions.

87

88　**2. Material and methods**

89　*2.1 Laboratory tests*

90　Different sorption materials for retaining PO_4-P were first studied in the laboratory in

91　batch (year 2010) and column (year 2011) tests. The materials studied in batch tests were

92　selected based on a literature review considering material cost-effectiveness, retention

93　efficiency, particle size, availability and costs. Locally available materials and by

94　products were carefully considered to avoid e,g. high transport costs and steel slag (basic

95　oxygen furnace slag (BOF) including mainly calcium oxide (CaO), Fe and silicon dioxide

96　(SiO_2)) from Raahe Rautaruukki Oyj (nowadays SSAB) was chosen. Filtralite®P (a clay

97   material with high Ca and Mg content), CFH 12 (ferrihydroxide), limestone (mainly

98     $CaCO_3$), Phoslock® (95% bentonite clay material + 5% lanthanum), and iron gypsum

99   from Kemira Oyj was also selected as promising materials. In these batch tests, runoff

100   from a peat extraction area (8 µg PO_4-P L^{-1}) and pore water from the peat or mineral layer

101   of TWs (40 and 250 µg PO_4-P L^{-1}) were used. The pH in these waters varied from 5.6 to

102   6.4, with the highest values occurring in runoff water. The batch test on each material was

103   performed with 100 g of sorption material and 200 mL of water in four parallel samples

104   (replicates), because ratio represent more realistically the field conditions. We followed

105   the protocol described by Heikkinen et al. (1995): the mixture of sorption material and

106   water was first shaken mechanically for one hour (shaker speed was selected between

107   300-400 min^{-1} in order to have all the tested mass be in circulation in the bottle), followed

108   by a 23-hour standing time. After this, the samples were shaken for 10 minutes and

109   centrifuged for 15 minutes at 4000 revolutions per minute. Finally, the samples were

110   filtered through a Whatman® GF/C filter (pore size 1.2 µm) and a Gelman filter (pore

111   size 0.45 µm). The samples were analysed for e.g. PO_4-P, pH, Al, Fe, Ca, Mg and sulphur

112   (S) in an accredited laboratory.

113   Column tests were run with steel slag (grain size 3-10 mm and 10-20 mm, which were

114   used in earlier studies also), CFH 12 and Sachtofer PR (iron gypsum material from

115   Sachtleben Pigments Oy) with peat extraction runoff water with an above-normal PO_4-P

116   concentration (650 µg PO_4-P L^{-1}). In the tests, two different-sized columns were used, so

117   that the diameter was at least 5 time higher than particle size (CEN ISO/TS 17892-11:fi),

118   but the needed water amount was minimized during test. In this way the difficulties in

119   collecting water and storing it were minimized. Columns with inner diameter 10.4 cm and

120   height 20.8 cm were used for steel slag with grain size 10-20 mm and Sachtofer PR, due

121   to their larger particle sizes, while columns with inner diameter 5.2 cm and height 31 cm

122   were used for steel slag with grain size 3-10 mm and for CFH 12. The height of these

123   sorption materials in the columns was about 15 cm. A filter paper (Whatman 114) was

124   installed in smaller columns and a filter mesh (pore size 0.5 mm) in larger columns to

125   prevent sorption material from ending up in the outlet pipe. In smaller columns, a quartz

126   sand layer (grain size 3-5 mm) was also added below (thickness 3 cm) and above (1.5

127   cm) the sorption material to ensure uniform water flow. Because the grain size of quartz

128   sand was <10-20 mm, this material was not used in larger columns. One column per

129   sorption material was used and the water was fed continuously by pump from the bottom

130   of these columns from 1 m^3 containers that were continuously mixed by a small pump.

131   The target outflow for small columns was 2 mL min^{-1} and for large columns 8 mL min^{-1}.

132   Thus the mean water residence time was calculated to be approximately similar (one hour)

133   in all columns. This residence time was used as it was possible to achieve also in field

134   conditions where the water discharge is high. However, the actual mean residence time in

135   columns varied from 1 hour to 1.5 hours and the flow velocity varied from 1.6 to 1.9 mL

136   min^{-1} in the small columns and from 5 to 8 mL min^{-1} in the bigger columns. When the flow

137   velocity was noticed to be clearly under the target outflow (2 or 8 mL min^{-1}), the pumping

138   speed was increased. These experiments were done in hydraulically saturated conditions

139   and run for almost three months (83 days).

140

141   The hydraulic conductivity (K) during the column testes was checked regularly to

142   determine clogging effects. First the K was first determined using empty columns where

143   was only filter mesh (large column) or filter paper and a quartz sand layer (small column).

144   The aim of that was to check how the column system restricted water flow (affected

145   hydraulic conductivity). After that, the hydraulic conductivity was determined for the filled

146   columns first one day after the beginning of the test, then approximately every week for

147   two first months, and finally at the end of the test. The hydraulic conductivity (K-value)

148   was calculated as (Tie- ja vesirakennushallitus, 1974):

149

150    $K = \frac{Q*L}{t*A*H}$ (1)

151

152   where Q is the amount of water (cm^3) flowing through the column sample at certain time t

153   (s), L is the height of sample (cm), A is the surface area of sample (cm^2) and H is the

154   pressure height (cm). Q was measured in the space without sorption materials on the top of

155   the column. On the occasions when hydraulic conductivity was measured i.e.

156   approximately every week for two first months, and finally at the end of the test, inflow and

157   outflow samples were also taken and analysed for PO_4-P, total P, pH, Ca, Al, Fe, S, Mg,

158   manganese (Mn), potassium (K), sodium (Na), barium (Ba), strontium (Sr), titanium (Ti)

159   and zinc (Zn) in an accredited laboratory. At the beginning of the test, samples were taken

160   from the water containers supplying inflow water for analyses of total nitrogen (N) (2300

161    $\mu g\ L^{-1}$), total P (670 $\mu g\ L^{-1}$), chemical oxygen demand (COD_{Mn}) (14 mg L^{-1}), suspended

162   solids (SS) (26 mg L^{-1}) and pH (6.5).

163

164   *2.2. In situ tests*

165   Steel slag and Phoslock® were also tested *in situ* in Äijönneva TW (64°5'13"N 25°4'56"E)

166   in northern Finland. Steel slag was selected because it is a cheap by-product, available

167   near the study site, can retain PO_4-P based on laboratory tests and does not leach sulphur.

168   Steel slag filters were constructed in the collection ditch as oxic drains to enhance P

169   retention. Slag was also placed in small heaps in front of perforated pipe holes, by request

170   of the peat extraction company who wanted to test this alternative. Phoslock® was selected

171   because it is known to retain PO_4-P well and has previously been used in lake restoration

172   projects. In lake restoration it is spread on the bottom of lake, where it binds PO_4-P from

173   the water column and the P released from the sediment. Phoslock® was spread on top of

174   the wet and partially water covered peat surface.

175

176   The Äijönneva area was drained some decades ago and is now covered with a dense tree

177   stand. The runoff water from the peat extraction area (surface area 103 ha) is pumped

178   through perforated pipes to the TW from June 2009 onwards. The wetland area (5.8 ha)

179   consists of two parts (southern and northern) separated by a main ditch called

180   Äijönkanava (Fig. 1). The average PO_4-P inflow concentrations were 20 µgL^{-1}, 50 µgL^{-1}

181   and 25 µgL^{-1} in summer 2009, 2010 and 2011, respectively. PO_4-P leaching from the TW

182   was observed during summer in these years, raising concerns as the environmental permit

183   expects P retention. The average daily outflow discharge from the TW, based on Finnish

184   Environment Institute hydrological watershed model system (WSFS; Finnish

185   Environment Institute, 2013), was 412 L d^{-1} in summer and 1220 L d^{-1} in autumn 2011.

186   The average inflow pH was 6.6, 6.6 and 6.5 and the average outflow pH was 6.0, 5.0 and

187   6.2 in summer 2009, 2010 and 2011, respectively. Other information on water quality and

188   purification efficiency results for Äijönneva TW can be found in Postila et al. (2014).

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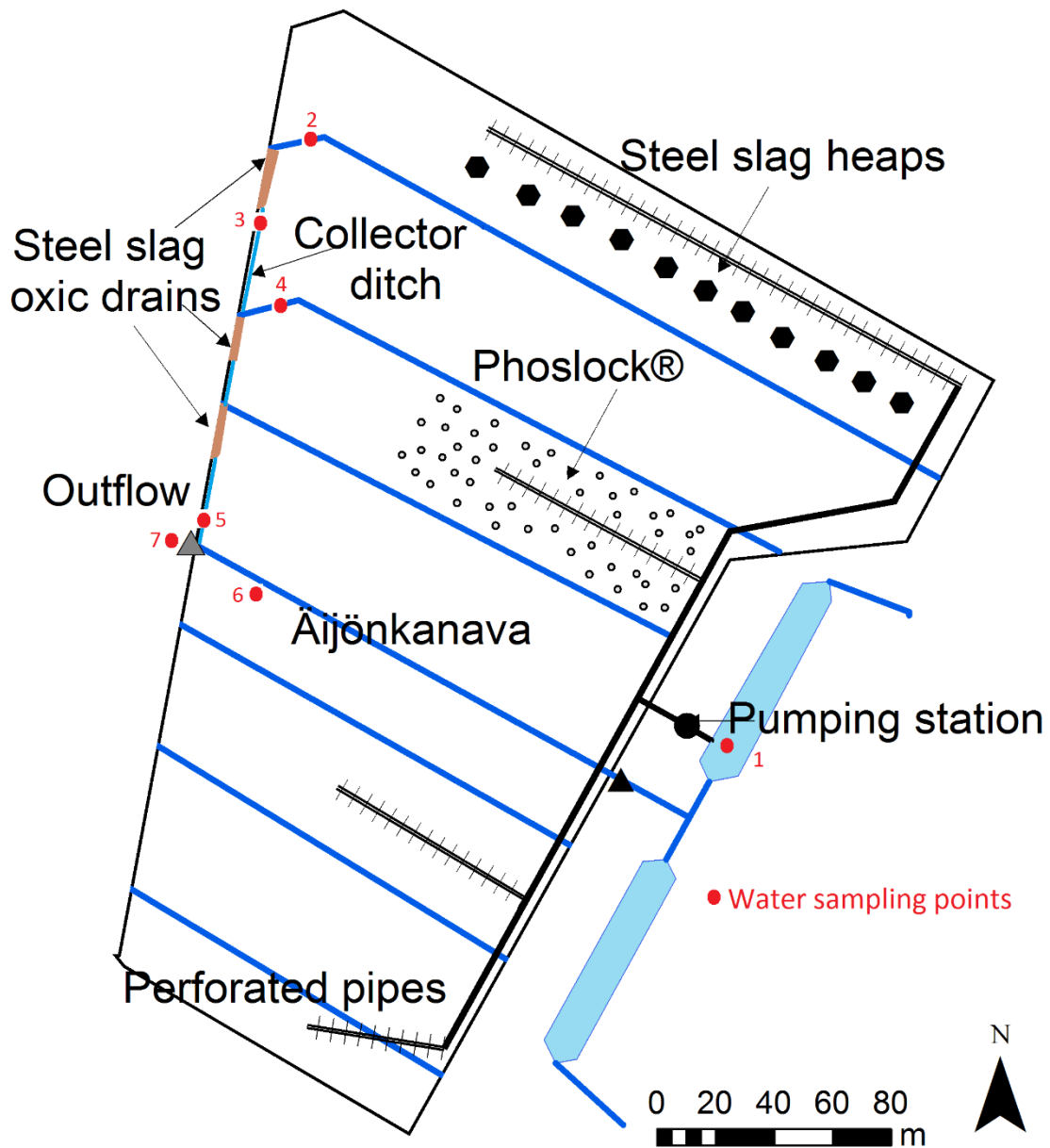
Fig. 1. Sketch of the Äijönneva wetland showing the position of perforated pipes and collector ditch, the locations of the two sorption materials tested (steel slag and Phoslock®) and the locations of water sampling points. The runoff water flows to the wetland via perforated pipes. On the wetland, the 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

197   the southern site of Äijönkanava channel, the water flows to the channel and finally to the

198   outflow point.

199

200   In late August-early September 2011, three steel slag oxic drains were constructed in the

201   collector ditch in the northern part of the wetland (Fig. 1). These oxic drains were 6-7 m

202   long and filled with 140 t steel slag with the grain size varying from 10 to 80 mm. The

203   grain size of steel slag used in oxic drains was larger than used in the laboratory test

204   (varying from 10 to 20 mm) as it was not possible to obtain large amounts of the finer-

205   grained steel slag used in the laboratory trial. Also ten tonnes of steel slag with grain size

206   7-15 mm were spread as small heaps in front of one perforated pipe outlet holes in the

207   northern part of the wetland because it was the other grain size, which was possible to get

208   field-scale amounts, and not so big than 10-80 mm. In addition, one tonne of Phoslock®

209   was spread around another perforated pipe.

210

211   Water quality was monitored in ditches below each test area and water flow outlets from

212   the control area (Fig. 1). The southern part of the wetland was then used as a control area,

213   because the water quality was quite similar to that in the northern part in autumn 2010. In

214   autumn 2011, water from all study points was analysed bi-weekly for pH, PO_4-P, total P

215   and dissolved total P in an accredited laboratory (Fig. 1). Wetland outflow water was

216   analysed once per month for Fe, Al, Mn, vanadium (V), cadmium (Cd), chromium (Cr),

217   copper (Cu), lead (Pb) and nickel (Ni), in order to monitor possible metal leaching from

218   the steel slag material. The water residence time in steel slag oxic drains was determined

219   using sodium chloride (8 kg NaCl in 150 L water) as tracer and measuring the changes in

220   electric conductivity in water.

221

3. Results and discussion

3.1 Batch tests

Based on the batch test results, all sorption materials studied can retain PO_4-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. PO_4-P retention (%) and mean values of water quality parameters measured after batch tests. Minimum and maximum values are given in brackets.

| Sorption material	PO_4-P retention efficiency (%)	pH	S (mg L^{-1})	Mg (mg L^{-1})	Fe (µg L^{-1})	Ca (mg L^{-1})	Al (µg L^{-1})
Steel slag	98-99	12.2 (12.1-12.2)	1.2 (1.1-1.5)	<0.1	18 (<5-23)	620 (580-660)	120 (98-150)
Filtralite®P	93-100	12.3 (12.2-12.3)	9.4 (8.2-12)	<0.1	6 (<5-13)	670 (660-690)	590 (530-640)
CFH 12	98-100	4.6 (4.4-5.5)	2700 (2600-2700)	1800 (1800-1900)	59 (41-87)	280 (260-300)	17 (13-21)
Limestone	70-93	7.9 (7.6-8.0)	0.81 (0.59-10)	3 (3-4)	2100 (1800-2500)	38 (28-50)	120 (29-210)
Phoslock®	88-99	6.4 (5.6-6.7)	16 (15-17)	20 (7-40)	<5 (<5-11)	170 (57-350)	2 (1-3)
Iron gypsum	98-99	7.7 (7.5-7.8)	610 (550-640)	63 (56-76)	17 (7-30)	530 (520-550)	30 (26-34)

234

235

236

237 *3.2 Column tests*

238 In the column tests, PO_4-P purification efficiency varied as a function of time (Fig. 2). As

239 expected based e.g. on Johansson (1999) and Xu et al. (2006), higher retention capacity

240 was observed for small particle-sized sorption materials (steel slag 3-10 mm and CFH

241 12). In addition, the Sachtofer PR iron gypsum material retained PO_4-P quite well except

242 at the end of the test, when PO_4-P purification efficiency was 0% and also Al, Fe, Mn, Zn

243 and Ti were leached. Typically, some decrease in the purification efficiency was observed

244 with time, caused by the cumulative P input and saturation, which can be expected (e.g.

245 Àdàm et al., 2007; Shilton et al., 2006; Søvik and Kløve, 2005). The difference in outflow

246 water pH between columns was greatest in the beginning of the test (Fig. 3). The pH of

247 water increased after contact with the steel slag material, which contains soluble CaO that

248 can elevate the water pH on dissolution (Klimeski et al., 2012). In the beginning of the

249 test, Sachtofer PR leached more than 600 mg S L^{-1} and CFH 12 over 200 mg S L^{-1}. The

250 Sachtofer PR column also leached S (160 mg L^{-1}) at the end of test. The Ca content in

251 water from the Sachtofer PR column was elevated during the whole test period. In the

252 columns of the other materials tested it was also elevated, but only in the beginning of the

253 tests. Total P, Al, Fe, Mn, Zn, Na, Ba, Sr, Mg, K and Ti concentrations in column outflow

254 were typically under or at the inflow level except for some individual samples, but Mn,

255 Sr and Mg concentrations in outflow from Sachtofer PR were generally elevated.

256

257 Hydraulic conductivity in the small particle size materials tested was lower than in the

258 larger particle size materials (Fig. 4), as previously observed by Penn et al. (2012). The

259 small particle size materials can also become clogged easily (Vohla et al., 2011). The

260 hydraulic conductivity of CFH was low during the test period. However, in the steel slag

261   (3-10 mm) column the hydraulic conductivity suddenly increased noticeably. This was

262   probably caused by development of a preferential flow path inside the column, and did

263   not represent the real hydraulic conductivity in the material. During the test period there

264   was some variation in hydraulic conductivity, which was probably partly caused by

265   clogging and dissolution of clogged material. For all test runs, the K-values observed

266   were smaller than for empty columns (including only filter mesh or filter paper and quartz

267   sand), where the conductivity was 0.05 cm s^{-1} (larger columns) and 0.08 cm s^{-1} (smaller

268   columns), so the equipment did not affect the sorption materials hydraulic conductivity.
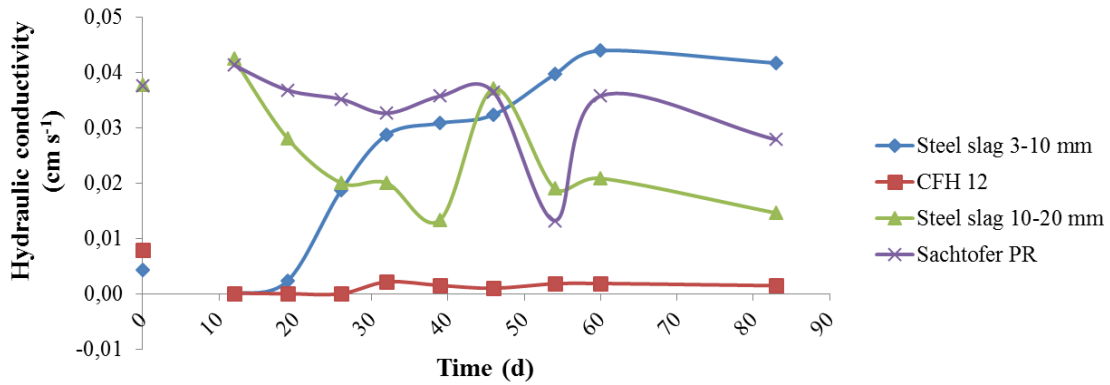
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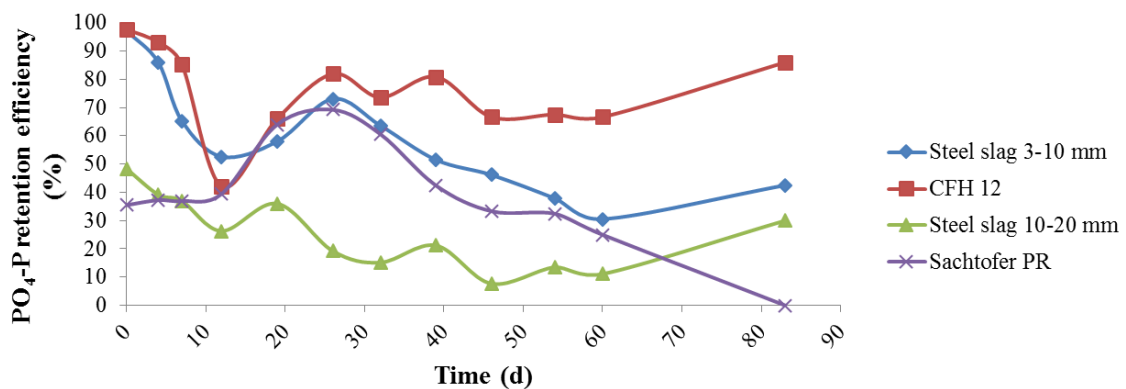
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271   Fig. 2. PO4-P retention (%) in different columns during the column tests.

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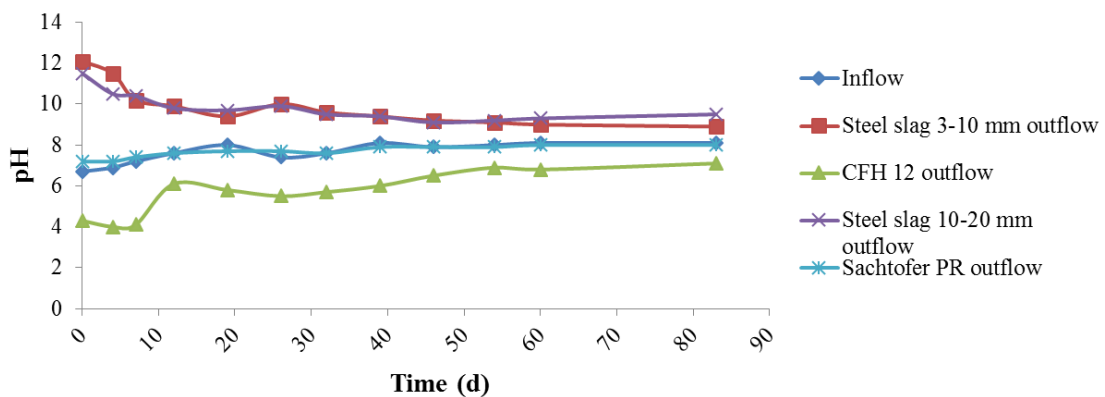
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274   Fig. 3. pH values in the inflow and outflow of different columns during the column tests.

275

276

277   Fig. 4. Hydraulic conductivity (cm s^{-1}) in different columns during the column tests.

278

279   *3.3 In situ tests in a treatment wetland*

280   Based on water samples from the TW, the constructed sorption systems did not retain

281   detectable amounts of PO_4-P (Fig. 5), total P or dissolved total P. In the ditch below the

282   Phoslock® area, the PO_4-P concentrations were sometimes smaller than in the other areas.

283   Moreover, the Cd, Cr, Cu, Ni and Pb concentrations were under the detection limit in all

284   outflow samples. The Mn and V concentrations were slightly elevated after the sorption

285   materials were installed, but their concentrations returned to their initial level by the end

286   of autumn at the latest. The Fe concentrations in outflow were lower than in inflow. Hence

287   there was no significant metal leaching from the sorption materials.

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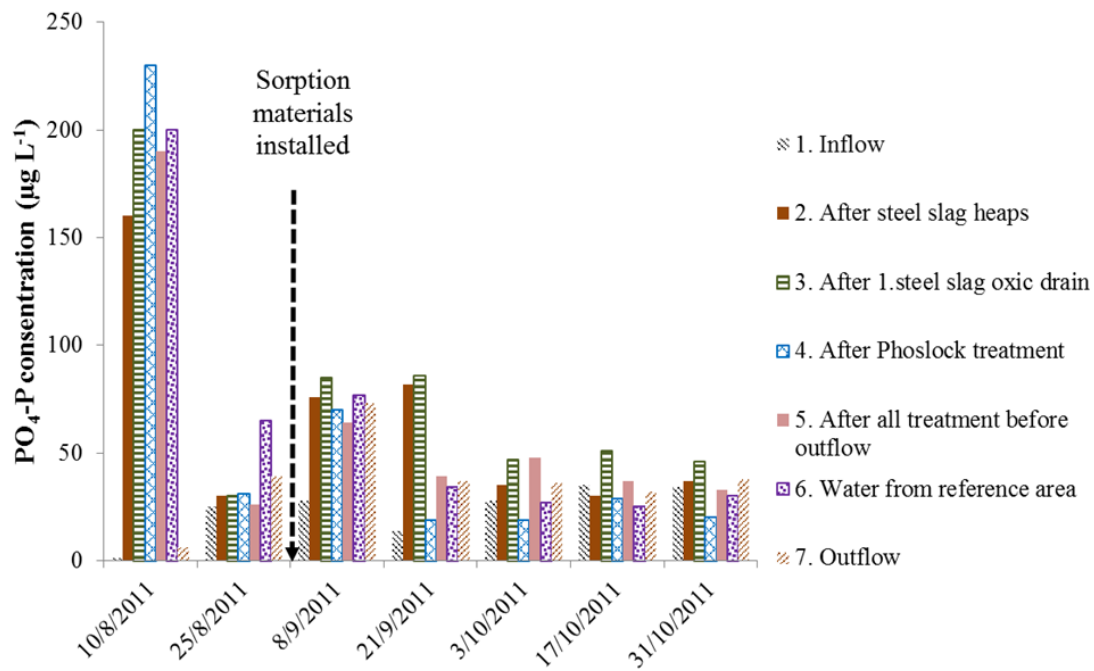
Fig. 5. PO$_4$-P concentrations (µg L^{-1}) in wetland water before and after sorption materials
were installed in Äijönneva wetland.

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

304 laboratory, but perhaps not in the field. When dissolved Ca^{2+} contact with atmospheric

305 CO_2 this can also lead formation of $CaCO_3$ precipitates (Drizo et al., 2002).

306

307 The small heaps of steel slag in the front of the perforated pipe holes may not have worked

308 for the same reason as the steel slag oxic drains. However, if these heaps are able to retain

309 some PO_4-P from inflow, it is possible that more PO_4-P is leached from the wetland peat

310 before the water flows to the wetland ditch. The Phoslock® material contains mostly clay,

311 which can harden during a dry period following a wet (rainy) period. Therefore it should

312 be spread as evenly as possible on the wetland surface. However, here it ended up being

313 applied in small heaps near one of the perforated pipes, the given instruction were not

314 fully understood and followed during construction works. Thus the Phoslock® formed

315 much less reactive surface area for P retention than could have provided if it were spread

316 evenly, as initially planned. As a result, this probably reduced the PO_4-P purification

317 potential of this material.

318

319 **4 Conclusions**

320 Sorption materials containing P-precipitating metals can be used for purification of

321 wastewater. We tested six different materials in laboratory experiments first in batch test,

322 and then selected the most promising materials for column tests runs (as Phoslock® is a

323 fine clay powder it was not used in column runs). Based on the laboratory test results and

324 considering steel slag as a cost-efficient by-product, steel slag and Phoslock® was tested

325 in field experiments. The results indicated that PO_4-P can be retained by sorption

326 materials under laboratory conditions, although due to cumulative P input into sorption

327 material columns, the sorption capacity can decrease over time. When steel slag and

328 Phoslock® were added to a treatment wetland, clear enhanced P purification was not

329 observed. The reasons for this may be too large grain size of the steel slag material used,

330 and consequently too short water retention time in this sorption material. In addition, the

331 pH of water passing through the steel slag was too low for Ca-rich sorption material to

332 precipitate P. It was concluded that sorption materials such as Phoslock®, which can be

333 spread on the wetland surface, should be evenly distributed to gain the most efficient P

334 sorption. Further studies are needed to identify suitable sorption materials and material

335 particle sizes for acidic field conditions with high hydraulic loading rate and low P

336 concentration. Overall, converting drained peatlands to efficiently working treatment

337 wetlands remains a challenge for research and water protection actions.

338

350

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