

LAB-SCALE COLUMN STUDY ON PHOSPHORUS REMOVAL FROM SYNTHETIC WASTEWATER BY *FILTRALITE P* AND IRON FILINGS

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ABSTRACT

Column study was performed in order to compare phosphate phosphorus (PO₄-P) removal capacity of iron filings and *Filtralite P*. The experiment with two vertical downflow columns (0.05 m in diameter and with 0.9 m medium height) feeding synthetic wastewater was carried out over a period of 66 days at wastewater temperature of 17.2–21.8 °C. The study also aimed to determine the effect of submergence of the medium on *Filtralite P* PO₄-P removal potential. During the experiment the submerged *Filtralite P* sorbed almost double amount of PO₄-P (1581 mg PO₄-P/kg filter material or 662 mg PO₄-P/m³ filter material) compared to the unsubmerged (881 mg PO₄-P/kg filter material or 369 mg PO₄-P/m³ filter material). In both cases PO₄-P removal efficiency exceeded 90 % when pH in the effluent was higher than 9.5. Through the experimental period the iron filings removed 2249 mg PO₄-P/kg filter material. When evaluating the amount of removed PO₄-P per volume of filter material, the iron filings removed 2164 mg PO₄-P/m³ filter material, i. e. 3.3 times more than the submerged *Filtralite P* did. In the case of iron filings the largest PO₄-P amount was removed in the top layer (0–30 cm) of the filter material. The amount of removed PO₄-P decreased and PO₄-P removal efficiency increased with depth of the medium: in the top layer (0–30 cm) PO₄-P removal efficiency was 27 %, whereas in the bottom layer (60–90 cm) it reached 44 %. The same tendency of PO₄-P removal efficiency was observed in the column with the submerged *Filtralite P*; however, the PO₄-P removal efficiency in all layers of this filter material was lower in comparison with the iron filings.

KEYWORDS

Wastewater, phosphorus removal, column experiment, *Filtralite P*, iron filings.

1 INTRODUCTION

Phosphorus is a fundamental element for normal functioning of ecosystems. Nevertheless, excess amount of phosphorus in water may cause a process of eutrophication, therefore the quantity of phosphorus that comes into contact with surface waters must be controlled [1].

Keplinger *et al.* (2004) [2] estimated that biological phosphorus removal by activated sludge and chemical phosphorus removal by application of reagents is not profitable for small communities. The alternative way of phosphorus removal is to use materials which compose of elements that could bind phosphorus. The scientists have investigated phosphorus removal by using various materials such as sand, crushed marble, vermiculite, calcite, limestone, alunite, shellsand, *Filtralite P*, *Polonite*, *Nordkalk Filtra P*, wollastonite, alum, blast furnace slag, electric arc furnace slag, ochre, iron filings [3–20]. These materials are divided into three groups: natural materials, industrial by-products and man-made sorbents [1, 21–23].

When phosphorus is removed using substrates, the phosphate phosphorus reacts with calcium, magnesium, iron and aluminium ions present in the substrate and forms insoluble compounds. Phosphorus removal capacity of man-made sorbents (e.g. *Filtralite P*, *Polonite*, *Nordkalk Filtra P*) is normally 10–100 times higher than the one of natural materials or industrial by-products [1]. On the other hand, an efficient substrate for phosphorus removal must not only possess a high sorption capacity but also have adequate hydraulic conductivity, physical chemical characteristics, recycling potential, reasonable cost and be locally available [24].

In real wastewater treatment systems, especially in Scandinavian countries, man-made sorbents *Filtralite P* and *Nordkalk Filtra P* are mostly used [22]. *Filtralite P* is particularly popular in Norway, the producing country. In Norway this substrate is usually used as a filter material in constructed wetlands [3]. *Filtralite P* has a high calcium and magnesium oxides (CaO and MgO) content. Exposed to water, these oxides form calcium and magnesium hydroxides, which, at certain pH value, split into Ca^{2+} and Mg^{2+} and these ions precipitate phosphorus [25]. There are some advantages of *Filtralite P* to be distinguished:

- high phosphorus sorption capacity measured in laboratory tests – Jenssen and Krogstad (2003) [26] have evaluated that *Filtralite P* phosphorus sorption capacity can reach up to 12 g P/kg filter material;
- the phosphorus saturated filter material can be used as a fertilizer in agriculture sector;
- high hydraulic conductivity;
- the substrate is highly studied with batch, column and field tests [24].

On the other hand, when using *Filtralite P* for phosphorus removal, pH in effluents can exceed the permitted discharge value and acidification or additional treatment of treated wastewater could be required. Moreover, *Filtralite P* is criticised for the high energy demand that the production processes consume and also for fairly high costs [1, 24].

Materials which remove phosphorus mostly by the interaction of the latter with iron present in the material are poorly studied. Kang *et al.* (2003) [27] have investigated phosphorus removal from secondary effluent by using laboratory produced minerals ferrihydrite, goethite and hematite. Heal *et al.* (2003) [10] have studied phosphorus removal from wastewater by ochre – a by-product from mine water treatment. Chung and Jeon (2000) [28] applied a method of anaerobic microbial corrosion for phosphorus removal. The principle of this method is that iron (iron nuts immersed in water, in this particular case) oxidizes when

exposed to the activity of sulphate-reducing bacteria. During the oxidation process ferric oxides are released, and then ferric oxides join phosphates to form slightly soluble compounds. Rolf *et al.* (1998) [19] and Burde *et al.* (2001) [6] have investigated the use of iron filings for phosphorus removal. The iron filings are a by-product of steel processing. The use of iron filings in wastewater treatment should be encouraged as a good practice model for the re-use of waste.

The objective of this study is to compare phosphate removal capacity of iron filings and *Filtralite P* during the column study and to evaluate phosphate sorption at different column heights. The study also aims to determine the effect of submergence of the medium on *Filtralite P* phosphate removal potential.

2 MATERIALS AND METHODS

The experimental set-up (Figure 1) consisted of two analogous structure vertical downflow columns filled with different filter medium – F1 (column with iron filings) and F2 (column with *Filtralite P*), two wastewater storage tanks (1 and 2) and a pump (3). During the tests the raw wastewater was stored in the bottom wastewater storage tank (1) from which it was transported to the upper wastewater storage tank (2) via the pump (3). From the upper storage tank the wastewater flowed by gravity into both columns, while even distribution of the influent was controlled by valves (4). The inner diameter of both columns was 5 cm, the height of the filter medium in the columns was 90 cm, thus the volume of both media was 1.8 l. The columns had interjacent wastewater sampling ports (6) that enabled sampling from different column height levels.

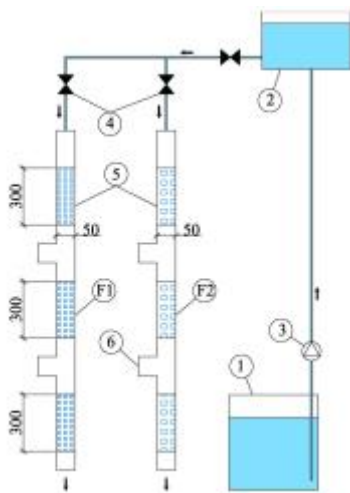


Figure 1. The schematic diagram of the experimental set-up: F1 – column with iron filings, F2 – column with *Filtralite P*, 1 – bottom wastewater storage tank, 2 – upper wastewater storage tank, 3 – pump, 4 – valve, 5 – filter medium, 6 – wastewater sampling port

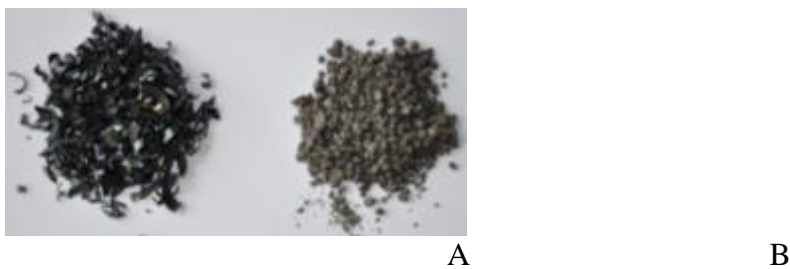


Figure 2. Tested media: A – iron filings, B – Filtralite P

The column F1 was filled with 1.7 kg of iron filings (Figure 2, A). The filings were of carbon steel (grade CT.45) and had the following chemical composition: 0.45 % carbon, 0.17–0.37 % silicon, 0.50–0.80 % manganese, <0.40 % chromium, <0.40 % nickel, <0.10 % molybdenum, <0.04 % sulphur, <0.04 % phosphorus. The size range of iron filings particles was 0.5–20 mm and bulk density was 963 kg/m³. The phosphate removal in the column with iron filings was based on chemical reactions – PO₄-P removal occurred when ferric oxides, generated during the oxidation process of iron filings, reacted with phosphate ions, thus forming slightly soluble compounds.

For comparison in the column F2 0.74 kg of man-made phosphorus sorbent *Filtralite P* (Maxit AS, Norway) was used (Figure 2, B). The *Filtralite P* consists of clay particles with a large specific surface area and a high porosity. It has a high pH and a high Ca and Mg content. The size range of tested *Filtralite P* particles was 0.5–4 mm. The *Filtralite P* bulk density was 370 kg/m³, particle density 910 kg/m³, particle porosity ~65 %, voids ~60 %, pH 12, alkalinity 35 mekv/l [29].

During the experiment iron filings were not submerged as oxygen was needed for the formation of ferric oxides. The column with iron filings was tested for 66 days. Aerobic conditions were not necessary when using *Filtralite P* for phosphate removal, thus, the effect of *Filtralite P* submergence on phosphate removal efficiency was analyzed during the experiment. For this purpose, the first half of the experiment (days 1–27) was run with the unsubmerged *Filtralite P*, while the second half of the experiment (days 28–54) was run with the submerged *Filtralite P*. In both cases the column with *Filtralite P* was fed with wastewater until phosphate removal efficiency of the sorbent became 0. When the filter medium was not submerged, the air to the columns was introduced by means of natural ventilation – the air circulated through columns inlet and outlet. When the medium was submerged, forced aeration was not applied.

The experimental set-up was fed with synthetic wastewater. It was prepared weekly by adding potassium hydrogen phosphate KH₂PO₄ into tap water. The characteristics of the wastewater fed to the columns are shown in Table 1. The wastewater flow rate was regulated every day of sampling. The columns were in operation on working days 10 hours a day, every column was supplied with 1 l/h of wastewater. Under these conditions hydraulic surface loading rate was 0.5 m³/m²/h.

During the first 54 days sampling of influent and effluent wastewater was done every working day. From day 55 (when the experiment was carried out only with the iron filings) the samples were taken twice a week. During the experiment days 32–44 seven samples were taken from each of interjacent sampling ports in order to analyze phosphate removal at different column height levels. All of the samples were grab samples and analyzed with

respect to phosphate phosphorus (PO₄-P), pH and dissolved oxygen (DO) concentrations. Chemical wastewater analyses were done according to the following methods:

- phosphate phosphorus concentration (PO₄-P) was measured by *Spectroquant* tests kits and *Genesys 10 UV-Vis* spectrophotometer (Thermo Fisher Scientific, USA);
- dissolved oxygen concentration (DO) and wastewater temperature were measured with oxygen meter *Oxi 330/SET* (WTW, Germany);
- pH was measured with pH-meter *pH 330i/SET* (WTW, Germany).

| | Average | Standard deviation | Lowest value | Highest value |
|--------------------------|---------|--------------------|--------------|---------------|
| PO ₄ -P, mg/l | 8.4 | 3.1 | 2.1 | 14.7 |
| pH | 8.1 | 0.3 | 7.1 | 8.5 |
| DO, mg/l | 8.2 | 0.4 | 7.3 | 8.7 |
| Temperature, °C | 17.2 | 21.8 | - | - |

Table 1. Characteristics of the wastewater fed to the columns

During the experiment PO₄-P removal efficiency of both tested materials was calculated according to the following equation:

$$E = \frac{C_{in} - C_{ef}}{C_{in}} \cdot 100, \quad (1)$$

here E – PO₄-P removal efficiency, %; C_{in} – concentration of PO₄-P in the influent of the column, mg/l; C_{ef} – concentration of PO₄-P in the effluent of the column, mg/l.

The amount of PO₄-P removed was calculated by the mass balance equation, assuming that the amount of removed PO₄-P is equal to the difference between the amount of PO₄-P in the influent and effluent of the column:

$$M = (C_{in} - C_{ef}) \cdot Q, \quad (2)$$

here M – amount of removed PO₄-P, mg/d; Q – flow rate, l/d.

3 RESULTS AND DISCUSSION

PO₄-P concentrations in the influent and effluent of the columns throughout the experimental period are shown in Figure 3.

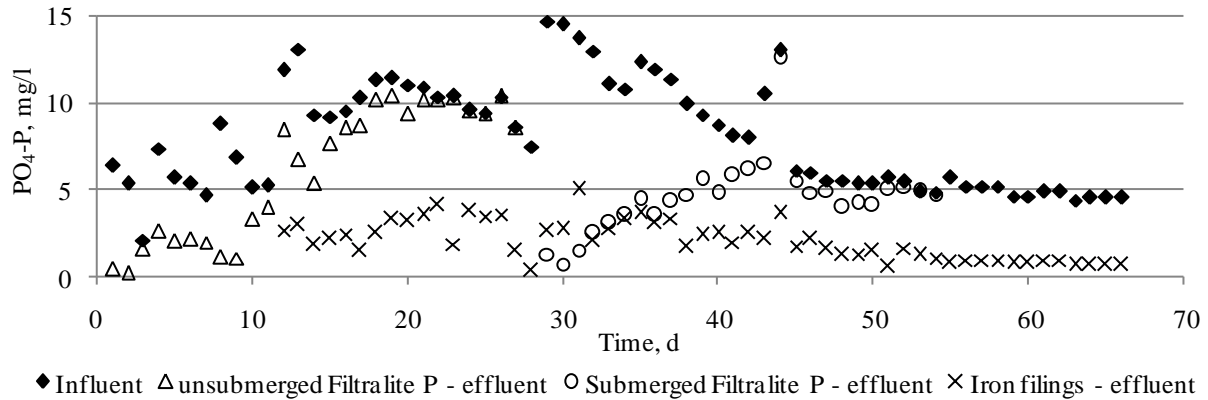


Figure 3. $PO_4\text{-P}$ concentration in the influent and effluent of the columns

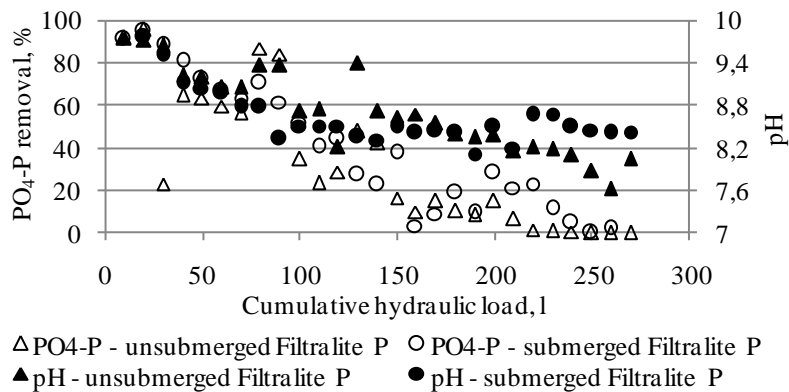


Figure 4. $PO_4\text{-P}$ removal efficiency and pH values in the effluent of the column with *Filtralite P*

Initially $PO_4\text{-P}$ concentration in the effluent was low in both cases: firstly, in the case of the un-submerged *Filtralite P* and, secondly, in the case of the submerged *Filtralite P*. In both cases $PO_4\text{-P}$ concentration increased gradually with time until it became equal to the $PO_4\text{-P}$ concentration in the influent. It might be seen from Figure 4 that *Filtralite P* $PO_4\text{-P}$ removal efficiency decreased over time as well as pH value in the effluent. In both cases initial pH value in the effluent was 9.8, however it decreased gradually to 7.6 in the case of the un-submerged *Filtralite P* and 8.4 in the case of the submerged *Filtralite P*. Whereas, in the effluent of the column with iron filings the pH remained stable at the value of 8.8 ± 0.2 (Table 2).

The pH of the *Filtralite P* medium is high due to calcium ions that react and bind phosphate ions. The experiments carried out by Adam et al. (2007) [3] using a column with *Filtralite P* and synthetic wastewater also had the same results: a gradual decrease in the effluent pH. The authors affirm that it must be a consequence of the calcium ions loss in the medium as it was also observed during the experiment. Figure 5 shows the correlation between *Filtralite P* $PO_4\text{-P}$ removal efficiency and the effluent pH. It can be seen that $PO_4\text{-P}$ removal efficiency was higher than 90 % when the effluent pH exceeded 9.5.

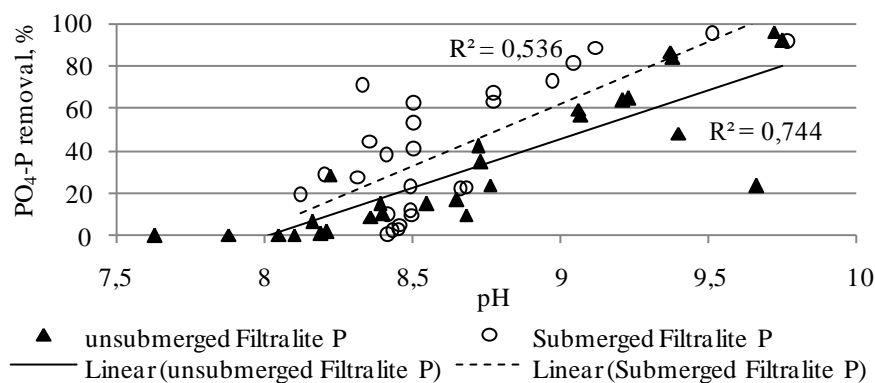


Figure 5. Correlation between the effluent pH and Filtralite P PO₄-P removal efficiency

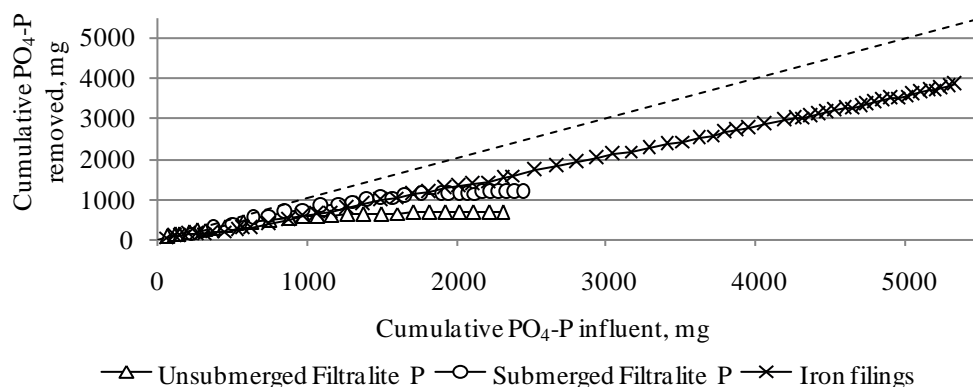


Figure 6. The cumulative amount of PO₄-P fed to the columns and the cumulative amount of PO₄-P removed

| | DO, mg/l | pH |
|----------------------------|----------|---------|
| Influent | 8.2±0.4 | 8.1±0.3 |
| Filtralite P – unsubmerged | 8.0±0.2 | 8.7±0.6 |
| Filtralite P – submerged | 8.4±0.2 | 8.6±0.4 |
| Iron filings | 7.2±0.6 | 8.8±0.2 |

Table 2. DO and pH values in the influent and effluent of the columns

In Figure 6 the cumulative amount of PO₄-P removed is compared to the cumulative amount of PO₄-P fed to the columns. Dashed line represents the case when the entire amount of PO₄-P fed is removed by the substrate. It can be seen that the submerged *Filtralite P* sorbs more PO₄-P than the unsubmerged substrate. However, in both cases *Filtralite P* gradually sorbs less PO₄-P and PO₄-P sorption curves in Figure 6 become horizontal. On the other hand, it is clear that while the *Filtralite P* almost did not sorbed PO₄-P, the iron filings kept on removing it successfully.

Table 2 shows that in the column with the *Filtralite P* DO concentration in the effluent was equal to the DO concentration in the influent. However, in the column with iron filings the DO concentration in the effluent was lower than in the influent. It is supposed that the decrease of DO concentration is related to the reaction between the iron ions in iron filings and the oxygen in the wastewater forming ferric oxides as a result.

From Figure 7 it is seen that PO₄-P removal process in the column with iron filings was stable when the PO₄-P volumetric load was in the range of 2.5–8.3 g PO₄-P/m³/h. With the increasing PO₄-P load, the concentration of PO₄-P in the effluent also increased, although PO₄-P removal efficiency was stable and ranged from 60 to 95 %. The average PO₄-P removal efficiency of the iron filings throughout the experiment was 69±16 % and the average PO₄-P concentration in the effluent was 2.4±1.1 mg/l.

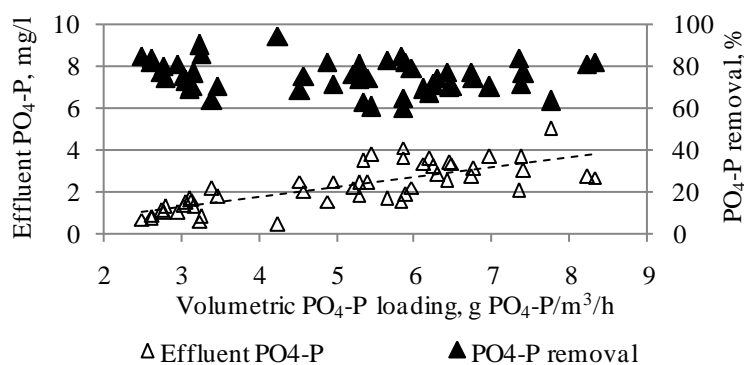


Figure 7. Effect of the volumetric PO₄-P loading on PO₄-P concentration in the effluent and removal efficiency of iron filings

| | <i>Filtralite P</i> ¹ | | Iron filings ² |
|--|----------------------------------|-----------|---------------------------|
| | Unsubmerged | Submerged | |
| Days of operation | 26 | 26 | 66 |
| Amount of fed wastewater, l | 260 | 260 | 660 |
| PO ₄ -P load, g | 2217 | 2392 | 5330 |
| Removed PO ₄ -P, mg | 652 | 1170 | 3823 |
| Removed PO ₄ -P, mg PO ₄ -P/kg filter material | 881 | 1581 | 2249 |
| Removed PO ₄ -P, mg PO ₄ -P/m ³ filter material | 369 | 662 | 2164 |

¹ Values showed in the table are obtained until the moment when PO₄-P removal efficiency became 0.

² Values showed in the table are obtained throughout the entire experimental period.

Table 3. Amounts of removed PO₄-P

The amounts of PO₄-P removed by both filter materials are shown in Table 3. The calculation of the PO₄-P amounts sorbed by *Filtralite P* includes phosphorus amounts sorbed until the PO₄-P removal efficiency became 0. Meanwhile the calculation of the PO₄-P amounts removed by iron filings includes phosphorus amounts removed throughout the entire experimental period. At the end of the experimental period PO₄-P removal efficiency of the iron filings was still high. In case the experiment continued, the iron filings would continue

PO₄-P removal from wastewater during a certain period of time and amount of removed PO₄-P would be even larger.

From Table 3 it is seen that *Filtralite P* sorbs almost double amount of PO₄-P when it is submerged in wastewater than when it is not. In the column experiment by Adam *et al.* (2007) [3] submerged *Filtralite P* sorbed 497 mg PO₄-P/kg material, which is 3 times less than in the experiment presented in this paper. This difference can be associated with variations in the experimental set-up such as PO₄-P concentration, hydraulic loading rate, structure of the column [24]. During the experiments by Adam *et al.* (2007) [3] the column of 0.14 m diameter was fed with 5.5 l/d wastewater flow rate. Through the experiment described in this paper the columns of 0.05 m diameter were fed with 10 l/d wastewater flow rate and higher hydraulic loading yields higher *Filtralite P* PO₄-P sorption [24].

At the end of the experiment the iron filings had removed 2.25 g PO₄-P/kg filter material, which is 1.4 times more than in the case of the submerged *Filtralite P*. On the other hand, it should be taken into account that bulk density of the iron filings is 2.6 times higher than the bulk density of *Filtralite P*. When evaluating the amount of removed PO₄-P per volume of filter material, it is seen that the iron filings have removed 3.3 times larger amount of PO₄-P than the submerged *Filtralite P*. Accordingly, three times less filter material is needed to remove the same phosphorus amount, which is an important advantage, because this kind of wastewater treatment system requires less space. Besides, it should be also taken into account that PO₄-P removal potential of the iron filings was not exhausted entirely during the experiment, thus, it can be assumed that they are able to remove even more phosphorus.

Figure 8 shows PO₄-P concentration in wastewater along the height of the columns, and Table 4 provides summarized results of the experiment.

From Figure 8 it is seen that PO₄-P concentration at different height of the column with iron filings is lower than of the column with submerged *Filtralite P*. Table 4 shows that in the column with iron filings amount of removed PO₄-P decreased with depth of the column, while the largest PO₄-P amount was removed in the top layer (0–30 cm) of the filter material. Adam *et al.* (2005) [25] while investigating *Filtralite P* material as well as Sovik and Klove (2005) [20] investigating shellsand in horizontal flow experimental systems, they both found that the largest phosphorus amount is accumulated at the inlet of the system. While as the distance from the inlet increases, the accumulated amount of phosphorus slightly decreases. Renman (2008) [18] and Gustafsson *et al.* (2008) [9] investigated phosphorus removal potential of various filter materials (blast furnace slag, opoka, *Polonite*, limestone, wollastonite, *Filtra P*) in vertical columns and also came to a conclusion that phosphorus is mostly removed in the top layer of the substrate. Whereas in real wastewater treatment systems distribution of phosphorus sorbed in filter material may vary. E. g., Zhu (1998) [30] found that the largest phosphorus amount is sorbed at the inlet of the system filled with light-weight aggregate, while Adam *et al.* (2006) [31] experiments showed that in subsurface flow wetland filled with *Filtralite P* material larger PO₄-P amount was sorbed at the outlet. In real wastewater treatment systems different distribution of phosphorus sorbed may be explained by possible differences of parameters such as hydraulic conductivity, particle size and porosity of filter material in different points of a huge system. Therefore PO₄-P sorption patterns established during column studies will not always mimic PO₄-P sorption patterns in real wastewater treatment systems [24].

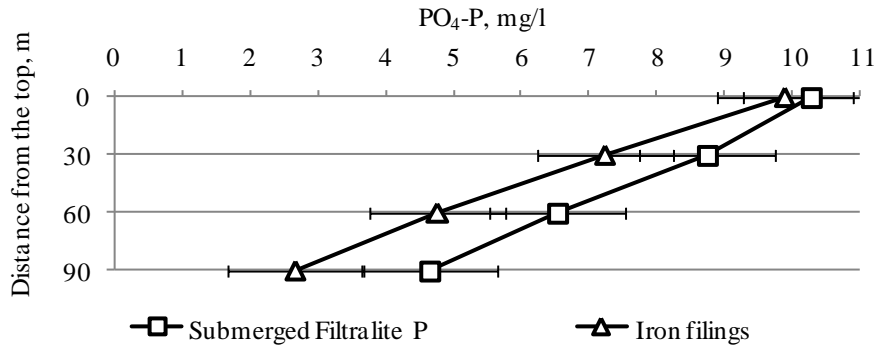


Figure 8. PO₄-P profiles at different column height

| Column height, m | Removed PO ₄ -P, % of concentration in the nfluent | | PO ₄ -P removal efficiency, % | |
|------------------|---|--------------|--|--------------|
| | <i>Filtralite P</i> submerged | Iron filings | <i>Filtralite P</i> – submerged | Iron filings |
| 30 | 27.2 | 36.8 | 14.9 | 26.8 |
| 60 | 39.4 | 34.1 | 25.4 | 34.0 |
| 90 | 33.5 | 29.2 | 28.9 | 44.1 |

Table 4. PO₄-P sorption at different column height

In the column with iron filings the PO₄-P removal efficiency increased with depth of the column (Table 4): in the top layer (0–30 cm) it reached 27 %, whereas in the bottom layer (60–90 cm) it was even 44 %. The same tendency was observed in the column with *Filtralite P*; however, the PO₄-P removal efficiency in all layers of this filter material was lower in comparison with the iron filings medium.

4. CONCLUSIONS

1. The correlation between *Filtralite P* PO₄-P removal efficiency and pH value in effluent from the column was established during the experiment: both in the case of submerged and unsubmerged *Filtralite P* the PO₄-P removal efficiency exceeded 90 % when pH in the effluent was higher than 9.5.
2. During the experiment the submerged *Filtralite P* sorbed almost double amount of PO₄-P compared to the unsubmerged *Filtralite P*: the submerged *Filtralite P* sorbed 1581 mg PO₄-P/kg filter material (662 mg PO₄-P/m³ filter material) until its PO₄-P removal efficiency became 0, while the unsubmerged *Filtralite P* sorbed only 881 mg PO₄-P/kg filter material (369 mg PO₄-P/m³ filter material). In both cases the sorption capacity of *Filtralite P* was higher than in the column experiments carried out by Adam *et al.* (2007) [3]. It can be associated with higher hydraulic loading rate as, according to [24], *Filtralite P* sorbs larger amount of PO₄-P at higher hydraulic loading rate.
3. Through the experimental period the iron filings removed 2249 mg PO₄-P/kg filter material, which is 1.4 times more than in the case of the submerged *Filtralite P*. When

evaluating the amount of removed PO₄-P per volume of filter material, the iron filings removed 2164 mg PO₄-P/m³ filter material, i. e. 3.3 times more than the submerged *Filtralite P* did. Moreover, it should be also taken into account that during the experiment PO₄-P removal potential of iron filings was not exhausted entirely, thus, it can be assumed that they are able to remove even more phosphorus.

4. In the case of iron filings the largest PO₄-P amount was removed in the top layer (0–30 cm) of the filter material. The amount of removed PO₄-P decreased and PO₄-P removal efficiency increased with depth: in the top layer (0–30 cm) PO₄-P removal efficiency was 27 %, whereas in the bottom layer (60–90 cm) it reached 44 %. The same tendency of PO₄-P removal efficiency was observed in the column with *Filtralite P*; however, the PO₄-P removal efficiency in all layers of this filter material was lower in comparison with the iron filings.

5. Iron filings are a promising material for PO₄-P removal from wastewater; however, further research is needed.

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