

# **Phosphorus Retention in Filter Beds for On-Site Wastewater Treatment: Improving the Design and Interpretation of Laboratory Filter Experiments**

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## **Abstract**

Filter beds of alkaline materials are a potentially useful technique to capture and recycle P in on-site facilities. However, it is unclear today how the investigation of new filter materials in the laboratory should be carried out in order to obtain reliable estimations of the materials' ability to retain P. The aim of this study was to formulate recommendations on how laboratory filter tests should be carried out. To investigate the effects of influent P concentration, influent type, loading rate and temperature on P retention in filters, three filter column experiments were carried out using 2<sup>2</sup> full factorial designs and four commercial filter materials. Statistical power was calculated to determine the required number of experimental replicates. The determined P binding capacity decreased when the influent P concentration increased and when wastewater was used as an influent instead of P solution. The determined P binding capacity increased when the temperature increased. Increasing the loading rate had an opposite effect on two filter materials. Particulate P was observed to be washed out of the experimental filters showing that it is important to measure both total and dissolved P in the effluent.

**Keywords:** Constructed wetland; Loading rate; Temperature, Concentration, Replicates

## **Introduction**

Eutrophication of natural waters is a global pollution problem. In Sweden, on-site wastewater treatment facilities contribute substantially to the nutrient pollution of the Baltic Sea (HELCOM, 2005) because of their insufficient ability to retain phosphorus (P). Filter beds of alkaline materials (Johansson Westholm, 2006; Vohla, Kõiv et al., 2011) are a potentially useful technique to capture and recycle P in on-site facilities. The filters are passive and robust and also a low-cost technique if cheap filter materials are available. Waste materials that have been tested for use as filter material include waterworks sludge (Zhao, Babatunde et al., 2009), sediment of oil shale ash, residues from the iron and steel producing industry and coal ash (Vohla, Kõiv et al., 2011), and ashes from the incineration industry (Johansson Westholm, 2006). Locally available materials need to be tested in laboratory prior to designing full-scale filters. However, it is unclear how the laboratory testing should be carried out in order to obtain reliable estimations of P retention in the filter materials.

This paper assimilates some of the results of a multiannual project on filter beds for capturing P in on-site facilities. The project aimed at increasing the understanding of P retention in P filters under varying conditions and at identifying measures for more reliable methods for testing filter materials in laboratory-scale. From this project, a PhD thesis (Herrmann, 2014) and several papers (Herrmann, Jourak et al., 2012; Herrmann, Jourak et al., 2013a; Herrmann, Jourak et al., 2013b; Herrmann, Jourak et al., 2014; Herrmann, Nordqvist et al., 2014) emerged. The paper at hand synthesizes the results of the investigation of four commercial filter materials: It was studied how a range of factors that commonly vary in the laboratory and/or at full-scale affect the P binding capacity of these materials as determined in laboratory column experiments. The aim was to formulate recommendations on how laboratory filter tests should be carried out.

## Material and methods

### *Materials*

The filter materials used in the experiments were Filtra P, Filtralite P, Top16 and Polonite, all being commercial products. The properties of the materials are given in were summarized by Herrmann (2014). Filtra P was manufactured from iron-containing gypsum and lime and was supplied by Nordkalk, Finland. The material is no longer available as the company stopped the production of the material ca. 2010. Filtralite P is derived from Norwegian lightweight aggregate and supplied by Weber Saint-Gobain Byggevarer, Norway. The Top16 material is manufactured by Envitop Ltd in Finland. Polonite is manufactured from Opoka rock in Poland that is heated to 900°C (Gustafsson, Renman et al., 2008) and is distributed by Biotech AB, Sweden.

### *Filter column experiments*

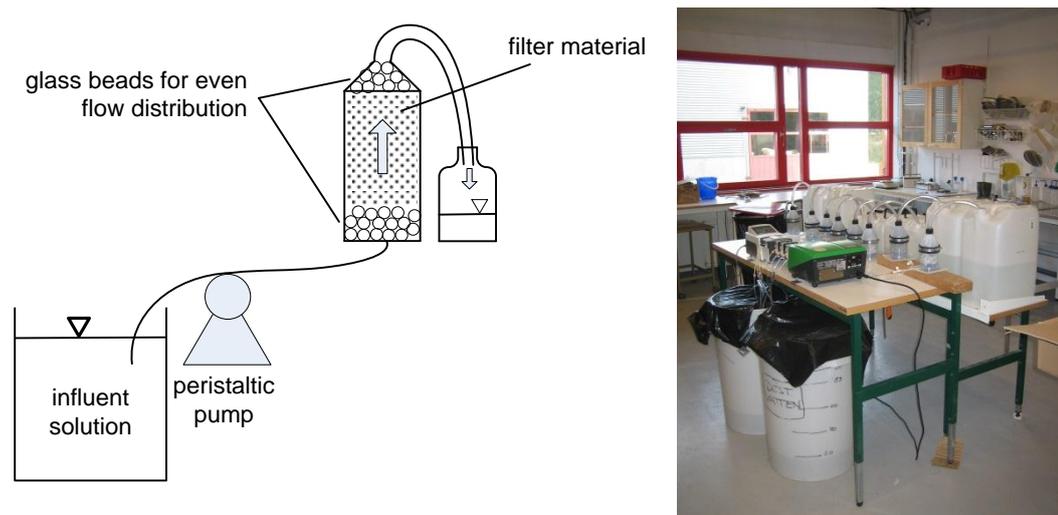
*Experimental design.* To investigate the effects of influent P concentration, influent type, loading rate and temperature on P retention in filters, three filter column experiments (experiment A, B and C) were carried out using 2<sup>2</sup> full factorial designs (Montgomery, 2009) with replicates (n = 2). In experiment B, three centre points were added while in experiments A and C, no centre points were included. The investigated factors and their settings are shown in Table 1. The low level of P concentration (12 mg L<sup>-1</sup>) reflected the typical concentrations in untreated on-site wastewater (Swedish EPA, 2006) and also resembled the P concentrations of secondary effluent in on-site facilities that have been found to be between ca. 5 and 14 mg L<sup>-1</sup> (Arias, Brix et al., 2003). The low level for loading rate (varying between 96 and 419 L m<sup>-2</sup> d<sup>-1</sup> across the three experiments) resembled the loading rates commonly present in compact full-scale filters i.e. in small filter units, typically filter wells. The high levels of P concentration (50 mg L<sup>-1</sup>) and loading rate (1023 (experiment A) and 1122 L m<sup>-2</sup> d<sup>-1</sup> (experiment B)) reflected extreme conditions aimed at accelerating the laboratory testing. The low level for temperature (4.3°C) resembled the operational conditions that are likely to prevail in full-scale filters for a substantial part of the year, particularly in areas with cool climates, while the high level for temperature (16.5°C) reflected conditions that are usually present during laboratory testing.

**Table 1** Investigated factors and experimental settings in the three filter column experiments

Variable	Experiment		
	A (Herrmann et al. 2014a)	B (Herrmann et al. 2013b)	C (Herrmann et al. 2014b)
Investigated factors	influent P concentration loading rate	type of influent loading rate	temperature material
Influent solution	P solution 12 mg P L <sup>-1</sup> P solution 50 mg P L <sup>-1</sup>	P solution 13 mg P L <sup>-1</sup> wastewater <sup>a</sup> 13 mg P L <sup>-1</sup>	wastewater <sup>b</sup> 12 mg P L <sup>-1</sup>
Loading rate [Lm <sup>-2</sup> d <sup>-1</sup> ]	419; 1023	96; 632; 1122	342
Temperature	room temperature	room temperature	4.3°C 16.5°C
Materials	Filtra P	Filtralite P	Top16 Polonite

<sup>a</sup>spiked with urine <sup>b</sup>spiked with K<sub>2</sub>HPO<sub>4</sub>

*Experimental set-up.* Using peristaltic pumps, the influent solution was passed in up-flow mode through the filter columns and the effluent was collected in 20L-plastic containers (Fig. 1). The columns were made of acrylic glass and had an inner diameter of 74 mm. A layer of glass beads was placed on the bottom of the columns to ensure an even flow distribution. The outlet of the columns was funnel-shaped and also filled with glass beads to ensure a smooth outflow (Fig. 1).



**Figure 1** Experimental set-up used for the three filter column experiments.

*Effluent sampling.* Twice weekly, the weight of the effluent was recorded and samples were taken for the analyses of total and dissolved P, total and dissolved organic carbon (TOC and DOC, only in experiments B and C), total suspended

solids (TSS), and for measuring the turbidity (only in experiment A). The pH and redox potential were measured from grab samples taken at the same time as the effluent samples. During sampling, the flow through the filters was interrupted for some hours.

*Data evaluation.* To determine the points of breakthrough and filter saturation, threshold values were defined for the ratio of effluent dissolved P to influent dissolved P. The point of breakthrough was defined based on an effluent P concentration of  $1 \text{ mg L}^{-1}$ , a figure normally used as the discharge limit for small-scale wastewater facilities in Norway (Heistad, Paruch et al., 2006) and also given as a guidance value by the Swedish Environmental Protection Agency (Swedish EPA, 2006). Data were further evaluated using analysis of variance (ANOVA) and multiple linear regression and the MODDE software package (Umetrics AB, 2006). The significance level was set to  $\alpha = 0.05$ .

*Power analysis.* Statistical power (the probability of rejecting a false statistical null hypothesis, eq. 2) was calculated retrospectively for the three column experiments to evaluate whether the effects of the factors on the total P binding capacities (calculated considering data from the entire breakthrough curves) of the investigated materials were reliably determined. The total P binding capacity comprises both the retained dissolved P and the retained or washed out particulate P i.e. the whole quantity of retained P for the wastewater columns and the whole quantity of reacted P for the P solution columns. The null hypotheses in the factorial experiments were that there was no effect caused by the investigated factors and their interactions on P binding capacity.

$$power = 1 - F(f_{\alpha}; 1, v, \lambda) \quad (2)$$

where:

power	probability of rejecting a false null hypothesis,
F	probability of the F distribution,
$f_{\alpha}$	critical value (upper point of the F distribution with 1 and v degrees of freedom) with $\alpha = 0.05$ ,
v	degrees of freedom for error, and
$\lambda$	noncentrality parameter, $\lambda = n * r * \delta^2 / (4\sigma^2)$ , where n is the number of replicates, r the number of corner points, $\delta$ the effect and $\sigma$ the estimated standard deviation.

For the calculations, the number of factors (two), the number of corner points (four) and the number of replicates (two) were predetermined by the experimental designs and were the same for all three experiments. The number of centre points was zero in experiments A and C, and three in experiment B. The significance level was set to  $\alpha = 0.05$ . The standard deviation ( $\sigma$ ) was the square root of the mean square error (MS error) which was estimated based on the collected data. MS error equals the error sum of squares divided by the error degrees of freedom (MS error =  $SS_{\text{error}}/df_{\text{error}}$ ). MS error was calculated using non-transformed data and omitting insignificant model terms. The minimum detected effect that was of interest was specified as 25% of the average total P binding capacity of the respective experiment. This effect is the difference between the low and high

factor level means of total P binding capacity. The Minitab software package (Minitab Inc., 2010) was used for the calculations.

## **Results and discussion**

All investigated parameters (i.e. influent P concentration, influent source, loading rate and temperature) affected the P binding capacity determined for the tested materials. Detailed results of these experiments can be found in Herrmann et al. (Herrmann, Jourak et al., 2013b; Herrmann, Jourak et al., 2014; Herrmann, Nordqvist et al., 2014).

### *Improving the design and interpretation of laboratory filter experiments*

*Method of collecting and sampling the effluent.* During filter column experiments, either the entire volume of effluent can be collected and sampled or grab samples of the effluent can be taken. These two options have considerable influence on the experimental set-up. In the filter column studies presented the entire volume of effluent was collected, weighed and sampled to ensure accuracy, control and reliability of the experiments. Collecting the entire volume of effluent ensured the accurate monitoring of the P mass flow through the filters and facilitated the creation of a mass balance. However, the practical restrictions on both the volume of effluent that could be handled and the number of samples that could be taken limited the size of the filter columns and thus the residence time. Grab sampling, on the other hand, would allow for bigger columns and thus longer residence time, because the effluent does not need to be collected. As grab sampling is less work and time-intensive, it would also allow for longer experiments. Although accuracy would decrease, grab sampling might be advantageous when testing filter materials because the flow regime in the filter columns could be designed to be more similar to that found in the field. This would certainly be important for the obtained results as the loading rate and / or residence time affected the P binding capacities of Filtra P and Filtralite P (Fig. 3).

*Size, diameter and length of the experimental filter.* The design of laboratory filter columns used in this study was a balancing act between an attempt to simulate full-scale conditions and the necessity of down-scaling for practical reasons. The size of the filter columns used in the experiments was rather small. The benefits of a small filter size are a shorter testing time, because the filter material becomes saturated faster, and a high controllability of the experiments because the entire volume of influent and effluent can be captured and sampled instead of taking grab samples. Better controllability leads to more accurate results. However, the properties of small columns differ considerably from full-scale conditions, especially in terms of their shorter residence time, also implying that the performance of small laboratory filters differ from their performance in the field, making laboratory results less reliable.

When debating the size of the experimental column it is also important to consider wall and channelling effects and dispersion phenomena. However, the effects of wall friction, maldistribution of porosity and unstable dispersion coefficients can

be minimised if the column diameter and length are large enough in relation to the diameter of the particles of the packed bed (Han, Bhakta et al., 1985; Winterberg and Tsotsas, 2000). The effect of a short residence time, however, is very important to carefully factor in when designing laboratory filters, as residence time directly affects the formation of Ca-phosphates in the filter and thus directly contributes to P retention. Therefore, the residence time in the filters should be as similar as possible to that of a full-scale filter, as far as practical restrictions allow.

*Flow mode.* Up-flow mode was used in the column experiments because the water flow against gravity was assumed to distribute small Ca-phosphate particles more evenly in the filter material by carrying them upwards. Further, the filter columns were continuously loaded to decrease the time needed for saturation of the materials. In full-scale applications, intermittent loading is applied which increases the residence time of the wastewater in the filter during loading breaks. An increased residence time might favour the precipitation of Ca-phosphates e.g. HAP. HAP needs time to form; Drizo et al. (2002) detected HAP on the surface of electric arc furnace slag used in a column experiment with a residence time of 0.5 days as opposed to an experiment with a residence time of 0.34 days, where no HAP was found. Thus, with continuous loading as applied this study, lower P binding capacities might be determined compared to intermittent loading.

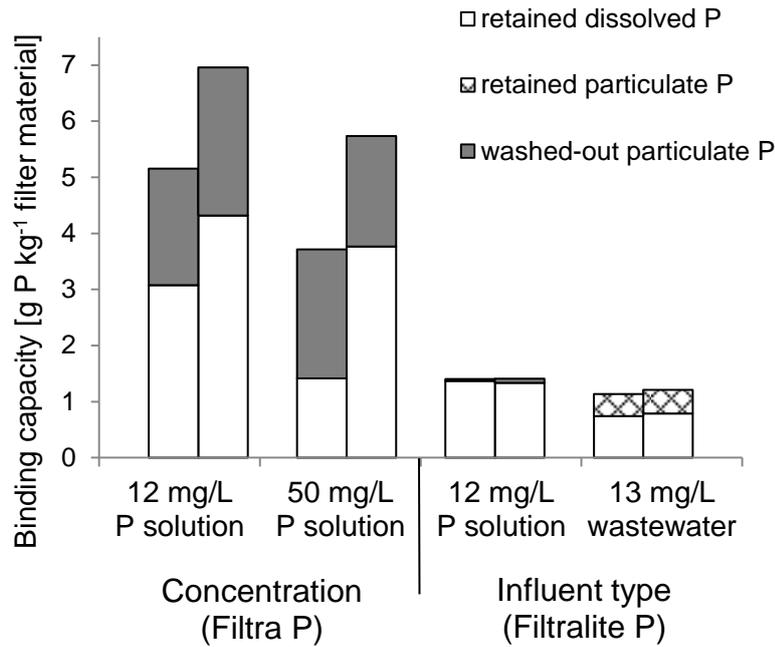
*Number of replicates.* The determination of statistical power (Table 2) showed a high value of power (1) for experiment C (Table 2) which means that the number of replicates was sufficient. Power was lower in experiment A (22%) and B (43%, Table 2). The probability of detecting an effect if it really existed was, therefore, rather low. In experiment B, significant factors were identified (the null hypothesis was rejected). However, in experiment A, the null hypothesis could not be rejected on the basis of the collected data as none of the investigated factors significantly affected the total P binding capacity (determined by considering data of the entire breakthrough curve). Thus, considering the low value of power, the result of experiment A cannot be regarded as reliable. The number of replicates that would have been necessary to obtain a power of 80% was 9 and 5 for experiment A and B, respectively. It should be noted that this number of replicates applies to a  $2^2$  factorial experiment i.e. the number of experimental runs would be 36. An experiment with this many replicates would therefore be almost impossible to undertake practically.

**Table 2** Statistical power of the filter column experiments A - C and the parameters used for its calculation. The factors that significantly affected the total P binding capacity in the respective experiment are also shown.

Parameter	Experiment (investigated filter material)		
	A (Filtralite P)	B (Filtralite P)	C (Top16, Polonite)
power	0.22	0.43	1
factors with significant effect	none	loading rate, influent type	temperature, material
terms omitted from the regression model in the power calculation	concentration, loading rate	interaction: loading rate×influent type	none
$\sigma = \sqrt{MS\ error}$	1.60	0.15	0.11
minimum effect [g P kg <sup>-1</sup> ]	1.6	0.2	1.1
number of replicates needed to obtain a power of 0.8 <sup>a</sup>	9 (actual power = 0.82)	5 (actual power = 0.87)	2 (actual power = 1)

<sup>a</sup>no terms omitted from model

*Influent properties.* Increasing influent P concentrations is a way of accelerating laboratory filter tests which has been practiced by several researchers (Drizo, Frost et al., 1999; Johansson, 1999; Drizo, Comeau et al., 2002; Drizo, Forget et al., 2006). However, in this study, an increased influent P concentration had a negative impact on the performance of the filters (Fig. 2): The total P binding capacity of Filtra P calculated using data from the entire breakthrough curve (until saturation) decreased when the influent P concentration was increased from 12 to 50 mg L<sup>-1</sup>. However, this difference in P binding capacity was not significant ( $\alpha = 0.05$ ). The higher P concentration lowered the pH regime in the filters. The pH is crucial for the precipitation of Ca-phosphates which is favoured at high pH (Feenstra and De Bruyn, 1979) and thus for the P retention in the filter. Decreasing the acid behaviour of the phosphate salt used in the solutions (KH<sub>2</sub>PO<sub>4</sub>) by adding a buffer to the influent might be a way of avoiding a low pH regime despite high P concentrations.



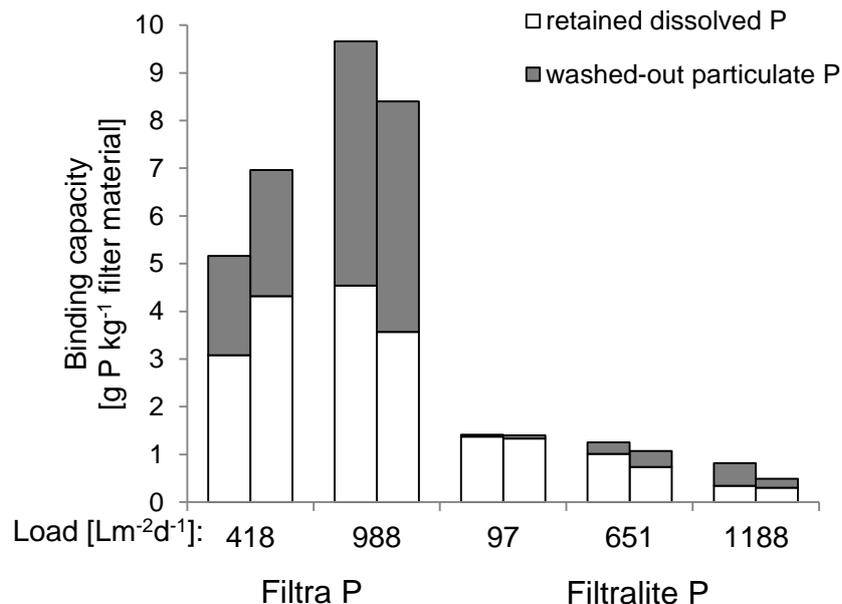
**Figure 2** P binding capacities of Filtra P determined at different influent P concentrations using P solution and an average loading rate of  $419 \text{ L m}^{-2} \text{ d}^{-1}$  and P binding capacities of Filtralite P determined using P solution and wastewater and an average loading rate of  $96 \text{ L m}^{-2} \text{ d}^{-1}$ ,  $n = 2$ . P binding capacities were calculated using data from the entire breakthrough curves.

Using secondary wastewater as an influent instead of P solution significantly decreased the P binding in Filtralite P (Fig. 2). Both the total P binding capacity and the amount of dissolved P retained in the filters were affected. The total P binding capacity comprises the retained dissolved P and the washed-out (P solution filters) or retained (wastewater filters) particulate P. Using secondary wastewater as an influent instead of P solution also significantly decreased the start pH and mean pH in the filters.

In laboratory studies, using synthetic P solution instead of wastewater would be more convenient but it would overestimate the P binding capacity determined because of the negative effect of wastewater. Adjusting the achieved P binding capacity with a correction factor would be a way of compensating for this effect. Based on the results of the Filtralite P experiments with the low loading rate, the correction factor would have to be 0.8.

*Concentration of organic matter and suspended solids.* Organic matter and suspended solids contained in the influent can potentially cause problems in the filter. Humic substances can combine with Ca ions (Song, Hahn et al., 2006), making them no longer available for the reaction with P. In addition, they can induce a clogging of the filter and a biofilm coating of the filter particles which would hamper the dissolution of Ca ions. It has been shown that higher BOD concentrations decrease P removal (Nilsson, Renman et al., 2013).

*Effect of loading rate on P binding.* In addition to an increased influent P concentration, loading rates higher than those in full-scale filters have been used to accelerate laboratory filter experiments (Brooks, Rozenwald et al., 2000; Seo, Cho et al., 2005; Ádám, Krogstad et al., 2005). In this study, increasing the loading rate had an opposite effect on P retention in Filtra P and Filtralite P (Fig. 3), showing that its effects are dependent on the material. In Filtralite P, the total P binding capacity significantly decreased when the loading rate increased (Fig. 3). The total P binding capacity of Filtra P increased with increasing loading rate, but this increase was not statistically significant. However, as the statistical power for the Filtra P-experiment (experiment A) was low (0.22, Table 2), there is a high probability (0.78) that there actually was an effect but it was not detected. Thus, there was at least an indication of a positive effect of the loading rate in the Filtra P data. Thus, increasing the loading rate to accelerate laboratory filter tests may give uncertain results because the effects of an increased loading rate are difficult to predict. The effects of the loading rate have not yet been comprehensively studied. However, a recent study shows that increased loading rates may negatively impact the retention of P in slags where the main retention mechanism is the formation of HAP crystals (Claveau-Mallet, Wallace et al., 2012).



**Figure 3** P binding capacities of Filtra P and Filtralite P determined at the different loading rates using P solution as an influent with total P concentrations of 12 mg L<sup>-1</sup> (Filtra P) and 13 mg L<sup>-1</sup> (Filtralite P)

*Loading rate versus residence time as a relevant design parameter.* Loading rate expresses the volume of fluid passing through the filter per time unit. The hydraulic residence time of the fluid in the filter column is calculated by dividing the pore volume of the filter column [L] by the flow [L d<sup>-1</sup>]. At a constant size of the filter column (i.e. constant pore volume), the residence time decreases with increasing loading rate. It can be debated as to whether loading rate or residence time is the more important parameter for P retention in the filters. Loading rate is the factor governing the pore velocity in the filter and can thus influence surface

reactions on the material as well as the fixation of Ca-P precipitates on the surfaces of the filter particles and between particles. Residence time, on the other hand, determines the time available for Ca-P precipitates to form and to form bigger particles e.g. HAP crystals, that can be mechanically retained in the filter more easily.

Two aspects lend weight to the argument to use loading rate as the relevant design parameter. First, in the materials used in experiment A and B, the amount of washed-out particulate P significantly increased with increasing loading rate, probably because the higher pore water velocity increased the transport of small P-containing particles towards the outlet of the columns. Second, it was shown in another study that the time needed for the formation of Ca-P compounds is short (Herrmann, Jourak et al., 2013b) and might therefore be of minor importance. Therefore, the residence times in experiments A - C were chosen to be rather low; they ranged from 40 minutes (experiment A) to 9.6 hours (experiment B).

However, the results obtained in this study showed that residence time was also an important parameter. Some effects observed when increasing the loading rate might have been due to the decreased residence time e.g. the decreased amount of retained dissolved P in Filtralite P (Fig. 3). It has been shown in previous studies that the formation of Ca-phosphate needs time. Drizo et al. (2002) reported that a residence time of at least 12 hours was needed for deposits of Ca-phosphate precipitates to occur on the surface of electric arc furnace slags and Mañas et al. (2012) found amorphous Ca-phosphate to be the precursor phase of HAP in granular sludge transforming to HAP in less than 7 days. The residence times associated with the investigated loading rates in experiment B were 9.3 hours, 83 and 46 minutes (Herrmann, Jourak et al., 2013b). Thus, possibly, a reduced precipitation of Ca-phosphates due to the decreased residence time might have caused the decrease in total P binding capacity and amount of retained P with increasing loading rate in experiment B. In addition, the escape of particulate P from the Polonite and Filtra P columns (Fig. 4) could partly be due to the residence time which might have been too short for these Ca-P precipitates to agglomerate or crystallize to particles that were big enough to be retained mechanically in the filter. Furthermore, P breakthrough in the filters occurred quickly for all investigated materials (Herrmann, 2014) although the loading rates were held at levels commonly used in full-scale filters, which indicates that the residence time was not sufficient and appears to play a major role in P retention. Therefore, the shorter residence time in laboratory columns compared to full-scale filters might implicate an underestimation of the determined P retention capacities.

*Ambient temperature.* Increasing the temperature from 4.3 to 16.5°C significantly increased the total and dissolved P binding capacity of Top16 and Polonite (see also Herrmann, Nordqvist et al., 2014). It also increased the average redox potential and the reduction of TSS, TOC and particulate organic carbon determined for both materials. The pH was not affected by temperature in either material. The results are discussed in detail by Herrmann, Nordqvist et al. (2014). As the P binding capacities determined were lower at the lower temperature, laboratory investigations carried out at room temperature might overestimate P retention.

*Particulate P in the effluent.* Both dissolved and total P concentrations were measured in the effluents of the experimental filter columns in this study. This appeared to be crucial because the total P concentrations in the effluents differed from the dissolved P concentrations (refer to Herrmann, 2014), showing that part of the P in the effluent was bound to particles. However, many previous studies focused on the soluble phase exclusively (Johansson, 1997; Brooks, Rozenwald et al., 2000; Cheung and Venkitachalam, 2000; Brix, Arias et al., 2001; Drizo, Forget et al., 2006; Gustafsson, Renman et al., 2008). In experiment A at both loading rates, and in experiment B at the high loading rate, the amount of P that was actually retained in the filters was of the same order of magnitude as the amount of washed-out particulate P (Fig. 11), thus stressing that the escape of particulate P merits critical attention.

## **Conclusions**

Secondary wastewater (despite varying properties over time) might be more suitable to be used as an influent in laboratory filter experiments because the use of P solution can overestimate P retention. For convenience, it is conceivable to use P solution instead of wastewater and correct for the effect with a correction factor. The organic matter content should be monitored and kept close to the figure expected in full-scale filters.

When increasing the influent P concentration to accelerate laboratory testing, one has to be aware of the consequences; the determined P binding capacity may decrease and breakthrough may occur earlier. The addition of a buffer to adjust the pH might be a way to compensate for these effects.

Increasing the loading rate affected P retention in Filtra P and Filtralite P, and especially increased the amount of washed-out particulate P. This effect should be considered when experimental loading rates are increased in the laboratory.

The size of the experimental columns should be maximised as much as is practical to avoid the adverse effects of a short residence time. Maximisation of the filter size should not be done at the expense of the column width.

The required number of filter replicates in experiments depends on the material investigated and the type of experiment carried out. The number of replicates required to obtain a power of 80% in the 2<sup>2</sup> factorial experiments A, B and C were 9, 5 and 2, respectively. These numbers can be a guidance for future experiments, but need to be recalculated if the experiment is of another type.

The placement of experimental filters in an ambient temperature resembling that expected in the field can be recommended.

It is important to analyse the concentrations of both dissolved and total P in the effluent of filter columns as there might be a considerable amount of particulate P in the effluent. Instead of collecting the entire volume of effluent, only taking grab

samples might be considered. However, this would be at the expense of mass balance calculations.

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