

Impact of hydraulic retention time on phosphorus removal from wastewater using reactive media

S. Benzing, F. Couceiro, S. Barnett, J. B. Williams, P. Pearce and C. Stanford

ABSTRACT

Phosphorus (P) discharge from wastewater treatment plants into the environment contributes to eutrophication issues. Reactive media filters represent an effective, simple and cost-effective solution to decrease the P content. Previous research used various experimental designs and often synthetic wastewater, making assessment of real-world performance difficult. This study assesses the impact of the hydraulic retention time (HRT) on P removal using real wastewater to refine design criteria for full-scale installations. Four media were compared in column experiments for >200 days. Different HRTs were applied and initially the media achieved low P effluent concentrations of >0.1 mg/L PO₄-P, increasing over time. Best P removal was observed for the highest HRT with on average >99%. HRT was seen to be the driving factor for P removal rather than media capacity. Three of the four materials showed pH levels above 12 initially, decreasing over time. Water quality parameters, including organics, solids and metals, were monitored. In-depth analysis confirmed formation of calcium phosphate precipitation on the media's surface. The results suggest the importance of an optimal HRT to achieve high P removal and show that the reactive media application is an appropriate technology for P removal on small sites if the elevated pH is addressed.

Key words | columns, phosphorus removal, reactive media, wastewater treatment

HIGHLIGHTS

- Systematic comparison of four reactive media types for P removal from wastewater.
- Long-term column studies with real wastewater at hydraulic retention times (HRTs) of 8–23 h.
- Effluents <0.2 mg/L PO₄-P over 200 days at HRTs >15 h, but effluent pH >12 an issue.
- Low HRTs (8 h) overloaded removal rates before final capacity reached.
- Reactive media filters a promising solution for small wastewater works if pH correction applied.

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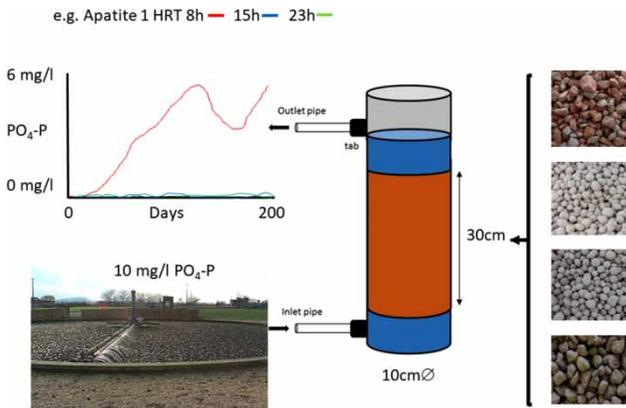
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GRAPHICAL ABSTRACT



INTRODUCTION

Nowadays the removal of phosphorus (P) from wastewater is of major concern to water management. The phosphorus discharged from sewage works into the environment contributes to increasing levels of eutrophication. As new phosphorus discharge regulations for small wastewater treatment plants (WWTPs) are expected, a need for cheap, more efficient and sustainable technologies arises (European Water Framework Directive 2000/60/EC). This study aims to assess P removal via reactive media in a filter bed application. Reactive media filters can be applied as a tertiary treatment on small WWTPs to reduce the P discharge to very low levels <1 mg/L PO₄-P via adsorption and precipitation processes. The reactive media system is a suitable alternative to chemical or biological P removal technologies due to their low maintenance and operational simplicity (Bunce *et al.* 2018). For more than a decade researchers all over the world have been investigating efficient, long-lasting and, at the same time, low-cost adsorbents (Callery *et al.* 2016). More than 100 materials (natural, industrial and man-made) have been tested for P removal in lab environments as well as field studies (Vohla *et al.* 2011). Various waste materials from industries, such as aluminium or steel making, have shown good P removal in previous research (Blanco *et al.* 2016; Cusack *et al.* 2018). Suitable media for this study were selected due to their composition, specifically in regards to their metal content, availability, cost, particle size and capacity. The chosen media were steel slag, calcium silicate bedrock and two types of granulated apatite as they were assumed to remove P to a very low effluent level and the output of previous trials were promising (Barca *et al.* 2014; Troesch *et al.* 2016; Rodríguez-Gómez & Renman

2017). Physical characteristics such as density and porosity vary between different materials and have to be accounted for in the determination of the size of the filter. Many previous studies were based on synthetic wastewater solutions and/or conditions, which can lead to misleading results due to the impact of wastewater characteristics, whereas this study utilises real wastewater and real-world conditions. Additionally, it is very difficult to compare the performance of the adsorptive materials in different studies due to highly variable experimental conditions such as P inlet concentration, hydraulic retention time (HRT), media particle size, wastewater characteristics, flow configuration and filter size. This study therefore provides direct comparison of different materials under the same conditions.

The purpose of this study is to evaluate the performance of four different filter materials for their ability to remove P by means of a column experiment mimicking a filter system in the field, and to further investigate the P removal mechanisms by means of X-ray powder diffraction (XRD), X-ray fluorescence (XRF) and scanning electron microscope (SEM) analysis. An optimal HRT which achieves high P removal is one of the most important parameters for the design of a full-size filter application. Since previous research has mainly investigated only one HRT, this study simultaneously examined three different HRTs for each of the media types. The selection of a suitable HRT determines the effluent P concentration, lifetime of the media and size of the filter system. In a second trial, triplicate columns were established at a suitable HRT selected by the results of the first experiment to test the P removal performance of the media at a higher concentration

and over a longer period of time. For one year, data was collected from the triplicates of each media in regards to soluble reactive phosphorus (SRP), total phosphorus (TP), biological oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS) and alkalinity.

MATERIALS

Four different commercially available materials were tested and their properties are shown in Table 1.

Steel slag is a by-product from the steel industry. Due to the abundance of steel slag and it being a waste product, the costs are low. The composition and characteristics of the steel slag media depend on the source and type of furnace it has been processed in. Steel slag from a basic oxygen furnace (BOF) was trialled in the first column study and slag from an electric arc furnace in the second. It primarily contains Ca and Fe oxides.

Two types of apatite (1 and 2) vary in the binder of the granulation process (the manufacturer did not reveal the specific binders used for commercial reasons). The analysis in Table 1 shows that apatite 1 had a higher percentage of CaO, which suggests a cementitious binder, and apatite 2 had a higher SO₃ content suggesting that some or all of the cementations material had been replaced, possibly with gypsum.

METHODS

The characteristics of the four materials and their P retention in column trials were studied.

Media properties

Mineral composition was evaluated via XRD analysis (Panalytical X'Pert3 Powder) and main oxides via XRF analysis (Panalytical Epsilon 4). Dry sieving determined the particle size. Bulk density was measured by the volume of water displaced by a known mass of medium. Porosity was determined from the amount of water needed to saturate a known volume of media. After 24 h suspension of 1:2.5 media (w): distilled water (V) the pH was recorded. All measurements were conducted in triplicates. Media samples before and after the column experiment were coated with carbon for SEM analysis (Zeiss EV0 MA10).

Columns

Bench-scale column experiments are the most commonly used method to predict pilot-scale design and operation parameters. To assess the selected media in this trial, a bank of 13 columns was established and experiments of varying HRTs were undertaken. Perspex columns with an inner diameter of 10 cm and a media height of 30 cm were setup as a saturated up-flow filter system. A constant flow rate was achieved by a 24-channel peristaltic pump. Columns were kept at room temperature. Filtered final effluent from the Petersfield WWTP (51°00'00.1"N 0°54'18.1"W), UK, was used as the inlet water and spiked to the desired P concentration of 10 mg/L PO₄-P with KH₂PO₄. Samples were taken from the column outlets and inlet.

Three different HRTs of 8, 15 and 23 h were tested for each of the four media, the vHRT (considering the void space) varied between the media due to their difference in porosity (39–56%). The columns were operated for a total

Table 1 | Media characteristics

	Ca-silicate bedrock	BOF slag	Apatite 1	Apatite 2
Main minerals	SiO ₂ , CaO	CaO, Fe ₂ O ₃	Apatite	Apatite
Main composition	SiO ₂ (55.2%) CaO (31.2%) Al ₂ O ₃ (6.3%) Fe ₂ O ₃ (4.0%) K ₂ O (1.4%)	CaO (30.6%) Fe ₂ O ₃ (28.9%) SiO ₂ (16.7%) Al ₂ O ₃ (11.2%) MnO (4.3%) Mg (3.4%) Cr ₂ O ₃ (2.3%)	CaO (65.1%) P ₂ O ₅ (24.8%) SiO ₂ (4.1%) SO ₃ (1.9%)	CaO (63.9%) P ₂ O ₅ (25.4%) SiO ₂ (3.6%) SO ₃ (2.5%) Al ₂ O ₃ (2.4%)
Particle size (mm)	2–6	2–10	2–6	2–5
Bulk density (g/cm ³)	0.8	1.7	1.4	1.2
Porosity (%)	56	39	39	50
pH	12.4	12.3	11.1	9.0

of 200 days with three breaks due to operational issues with pumps, between day 60–75 and 110–124 and 159–185 in which the columns were drained during the first break and kept saturated during the two others.

A second trial was conducted under the same conditions with an increased inlet SRP concentration of 13 mg/L and a HRT of 15 h. Columns were established in triplicates for Ca-silicate bedrock, apatite 1 and steel slag (apatite 2 was no longer available at that stage). The columns were operated and sampled for a year and stopped for 12 days at day 164–176 when they were drained.

Analysis methods

SRP analysis expressed as $\text{PO}_4\text{-P}$ was performed according to the Molybdenum Blue Method using the Autoanalyser 3 (Seal Analytics). The pH and water temperature were measured with the pH meter 3310 (Jenway). BOD_5 was determined according to *Methods for the Examination of Waters and Associated Materials* (Standing Committee of Analysts 1988). COD was analysed according to the Closed Reflux Colorimetric Method using the Palintest. Measurement of TSS was conducted via filtration. TP and alkalinity were determined via the colorimetric method using the Palintest. Metals were analysed by Inductively Coupled Plasma Optical Emission Spectrometry (Spectroblue). All of the analysis, unless otherwise stated, followed methods according to *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association 2012).

Samples for $\text{PO}_4\text{-P}$ and pH analysis were taken from the column inlet tank and outlets three times a week during the first column study and biweekly for the second trial. BOD and TSS were measured weekly and COD, TP and alkalinity fortnightly. Metals were analysed fortnightly for the first 90 days of the experiment ($n = 5\text{--}6$).

RESULTS AND DISCUSSION

First column trial: P removal and pH over time

All four media initially removed $\text{PO}_4\text{-P}$ to levels below 0.5 mg/L (Figure 1). Concentrations of >1 mg/L $\text{PO}_4\text{-P}$ were received after 20–40 days of operation from the column outlets containing Ca-silicate bedrock, steel slag and apatite 1 at the low HRT. Apatite 2, however, still showed good P removal after 90 days and is therefore the best performing media for P removal efficiency at high flow rates. The column effluent P concentration started to

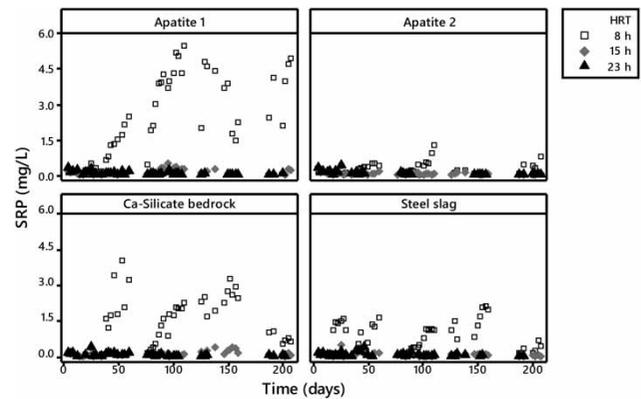


Figure 1 | SRP concentration of the column effluents for different HRTs for each of the media.

increase at the lower HRT of only 8 h, whereas the concentrations at higher retention times were still below 0.5 mg/L at that point. Therefore, the higher the HRT, the better the P removal of the media. This is supported by other studies, for example Barca et al. (2013).

After the 2 week resting period at day 60–75, the performance of the media at 8 h HRT, which previously showed an increase in the outlet P concentration of the columns, improved for a short period of time. At the same time, the pH increased (Figure 2). Enhanced P removal following a regeneration period of the media was also observed by Bird & Drizo (2010) and Karczmarczyk et al. (2019). Drizo et al. (2002) suggests the increase in P removal performance can be explained by new P adsorption sites due to the transformation of minerals to more amorphous forms resulting from a decrease in redox potential. Also, metal ions such as Ca, Fe and Al could have moved to the surface of the media during the resting period due to an increase in pH which created supersaturated conditions. Media recovery

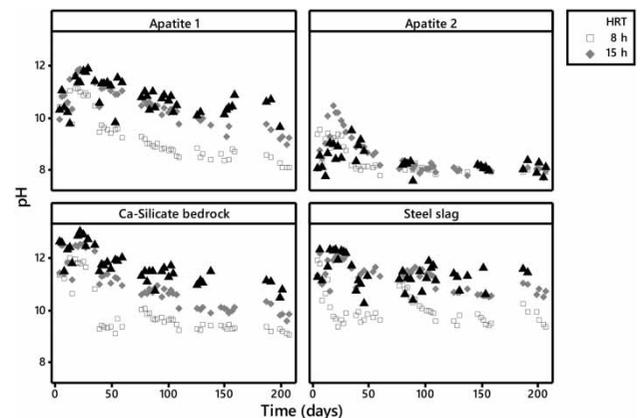


Figure 2 | pH of the column effluents over time for the different HRTs for each of the media.

is also seen after the second and third break (day 124 and 185), but to a lesser extent. Although new adsorption sites can be generated during the resting period, the amount of adsorption sites will always be less than at the start of the experiment, which means the media cannot be fully regenerated and the P uptake will still decrease over time.

All four media elevated the pH. The inlet water showed an average pH of 8.2 ± 0.2 . Values for the columns filled with Ca-silicate bedrock, steel slag and apatite 1 increased the pH to 10–12.5, whereas apatite 2 seemed to only have a small impact on the pH, elevating it to 8.4 on average. Highest pH values were recorded at the beginning of the experiment and there was a trend of decreasing pH with time in all cases. This is explained by less CaO dissolution over time due to a decrease in CaO availability and clogging of the surface due to precipitation, solid deposition and biofilm development (Troesch et al. 2016). In terms of real-world application, 200 days is still regarded as the initial phase, considering a lifetime of several years and the pH is expected to drop further after the first few months (Barca et al. 2013).



The HRT influences the pH. The longer the contact time, the higher the pH, as the media has more time to release OH^- into the water by CaO dissolution (Barca et al. 2014).

For the design of a full-size filter system, a high HRT means better P removal, however, it also requires a greater volume of media. A larger filter bed is therefore needed and the resulting effluent pH is higher.

Since the pH is elevated above 9, there is a need for a pH correction step after the media application in order to decrease the pH for the discharge into the environment. Preferably, this is achieved by a passive system such as wood bark filters or aeration.

The removal efficiency was calculated using the following equation:

$$\text{Efficiency: P (\%)} = \frac{C_{\text{in}} - C_{\text{out}}}{C_{\text{in}}} \cdot 100 \quad (2)$$

where C_{in} is the inlet and C_{out} the outlet concentration of the column in mg/L $\text{PO}_4\text{-P}$.

P removal rate

The HRT is a crucial factor for the P removal performance of a media. The higher the contact time between the P in the

water and the media, the more P can adsorb to the media's surface and/or precipitate. At the beginning of the experiment, up to a load of 0.3–1 mg P/g media, depending on the media (Figure 3), >99% of the $\text{PO}_4\text{-P}$ was removed due to the high CaO dissolution rate at the start (a high pH was observed) and there was plenty of free adsorption sites on the surface of the media. Over time, after 0.3–1 mg P/g media uptake, the removal decreased to >90% for the columns running at a low HRT of 8 h, which was accompanied by a decrease in pH of <10. However, at the same P uptake, the columns with a 15 h retention time still removed P to >97%–99% at a pH level of 10–11.6. This leads to the assumption that HRT determines the rate of P adsorption and precipitation rather than media capacity.

Metal removal/leaching

Metals were analysed to evaluate if the media leaches any metals into the water and/or if the media is able to remove any additional elements to P. All four media showed a reduction in Fe from the highly concentrated influent due to ferric dosing on site. Concentrations of Zn and Pb were significantly less at the outlet of the columns, with the exception of the Pb concentration in the 7 h column filled with apatite 2. A study on steel slag and Ca-silicate bedrock by Renman et al. (2009) also observed uptake of Cu, Mn, Pb and Zn by the two materials, which confirms the findings from this research. All of the media released Ca, especially at the beginning of the experiment, which coincided an increase in pH levels due to the CaO dissolution. Some Si and Cr leaching was also observed (Table 2).

All metal concentration values were below the standards for drinking water, according to the Council Directive 98/83/EC (European Commission 1998), with the exception of Cd and Pb. Cd leaching from the steel slag and apatite 1 (8 h HRT) exceeded the 5 µg/L threshold and the Pb concentration (>10 µg/L).

Metal retention onto the media and leaching must be considered for the potential use as a slow release fertiliser on agricultural land after its WWTP application, as it

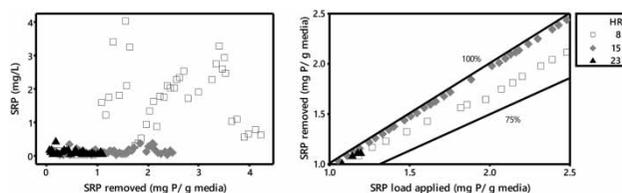


Figure 3 | P removal and load applied during the 6 months column experiment for Ca-silicate bedrock for the different HRTs.

Table 2 | Total metal concentration of the column effluents and inlet

	Inlet	Ca-silicate bedrock		BOF slag		Apatite 1		Apatite 2	
HRT		8 h	23 h	8 h	23 h	8 h	23 h	8 h	23 h
Ca (mg/L)	48.6	72.8	159.8	144.2	110.7	113.1	98.4	87.6	64.9
Cd ($\mu\text{g/L}$)	4.6	4.0	4.0	8.6	12.5	8.8	4.6	5.1	5.2
Cr ($\mu\text{g/L}$)	2.86	4.9	10.0	9.1	16.5	8.9	30.4	4.9	18.5
Cu ($\mu\text{g/L}$)	10.4	6.6	2.3	10.5	13.2	13.8	5.7	51.5	39.7
Fe ($\mu\text{g/L}$)	428.5	161.5	22.2	212.7	96.9	226.1	43.7	200	90.9
Mg (mg/L)	3.2	4.4	0.4	0.02	2.9	1.1	1.6	0.9	5.2
Mn ($\mu\text{g/L}$)	5.9	3.9	2.1	5.5	18.1	5.1	2.4	2.5	3.8
Ni ($\mu\text{g/L}$)	4.5	4.0	4.4	6.9	9.4	7.2	3.7	5.1	4.0
Si (mg/L)	0.6	10.6	7.1	0.8	12.4	4.8	6.6	6.0	1.8
Pb ($\mu\text{g/L}$)	13.7	11.3	11.4	9.8	7.4	8.6	5.0	15.9	8.4
V ($\mu\text{g/L}$)	<LOD	11.2	15.1	19.5	32.0	18.1	9.2	40.8	34.5
Zn ($\mu\text{g/L}$)	45.4	26.8	5.5	25.6	20.3	12.2	7.2	27.0	15.7

Note: LOD, limit of detection for V is <2.4 $\mu\text{g/L}$

could leach the accumulated metal back into the soil and therefore the food chain.

SEM

SEM images show the difference in morphology on the media surface before and after the first column experiment. Spherical-shaped precipitation can be seen on the surface of apatite 1 after the trial (Figure 4). Multiple spot samples analysed by energy-dispersive X-ray spectroscopy (EDX) determine the formation of calcium phosphates on the surface of steel slag and apatite 1, and the presence of P on the Ca-silicate bedrock after the experiment compared to the absence of P before. The P on the surface of apatite 1 is naturally occurring in the apatite raw material and was much higher after the trial. Additionally, as found by Barca *et al.* (2014), P can be incorporated into CaCO_3 precipitation on the surface, which cannot be detected by EDX in this study due to coating with C.

Second column trial

All media removed SRP to very low levels <0.2 mg/L $\text{PO}_4\text{-P}$ initially but the P removal efficiency decreased over time (Figure 5), which complies with results obtained from the first column study.

The media effluents exceeded 2 mg/L $\text{PO}_4\text{-P}$ after 254 days, 140 days and 163 days for Ca-silicate bedrock, steel slag and apatite, respectively. After 282 days the

removal was still quite high (62–84% depending on the media), but at that point the HRT was increased to 28 h to investigate if the P removal performance would improve. For all media the effluent concentration decreased to below 2 mg/L for the remainder of the experiment, >80 days. This again highlights that high effluent concentration is not due to the media's capacity but is dependent on HRT.

Along with an increase in HRT, a higher pH was expected due to results from the first column study. However, when the HRT was doubled no significant increase in pH could be seen (Figure 6). The improved P removal efficiency can therefore not be explained by an increased CaO dissolution as the pH stayed low. This leads to the assumption that the higher HRT favours increased P adsorption onto the media and possibly onto CaCO_3 , as found by Barca *et al.* (2014). On the one hand, carbonate from the water (high alkalinity from the inlet water, with a mean of 122 mg/L CaCO_3) competes with phosphate for adsorption sites and free Ca^{2+} , but on the other hand it can provide additional adsorption sites for P, and some P can be incorporated in CaCO_3 formation.

The main parameter monitored was SRP as the media mainly removed the soluble reactive P, whereas legal discharge requirements are usually expressed in TP. The SRP/TP relation was between 0.8 and 0.9. BOD_5 levels were very low in all cases and the columns slightly decreased COD levels. TSS were also low. The inlet alkalinity on site was quite high and was further increased by the media (Table 3).

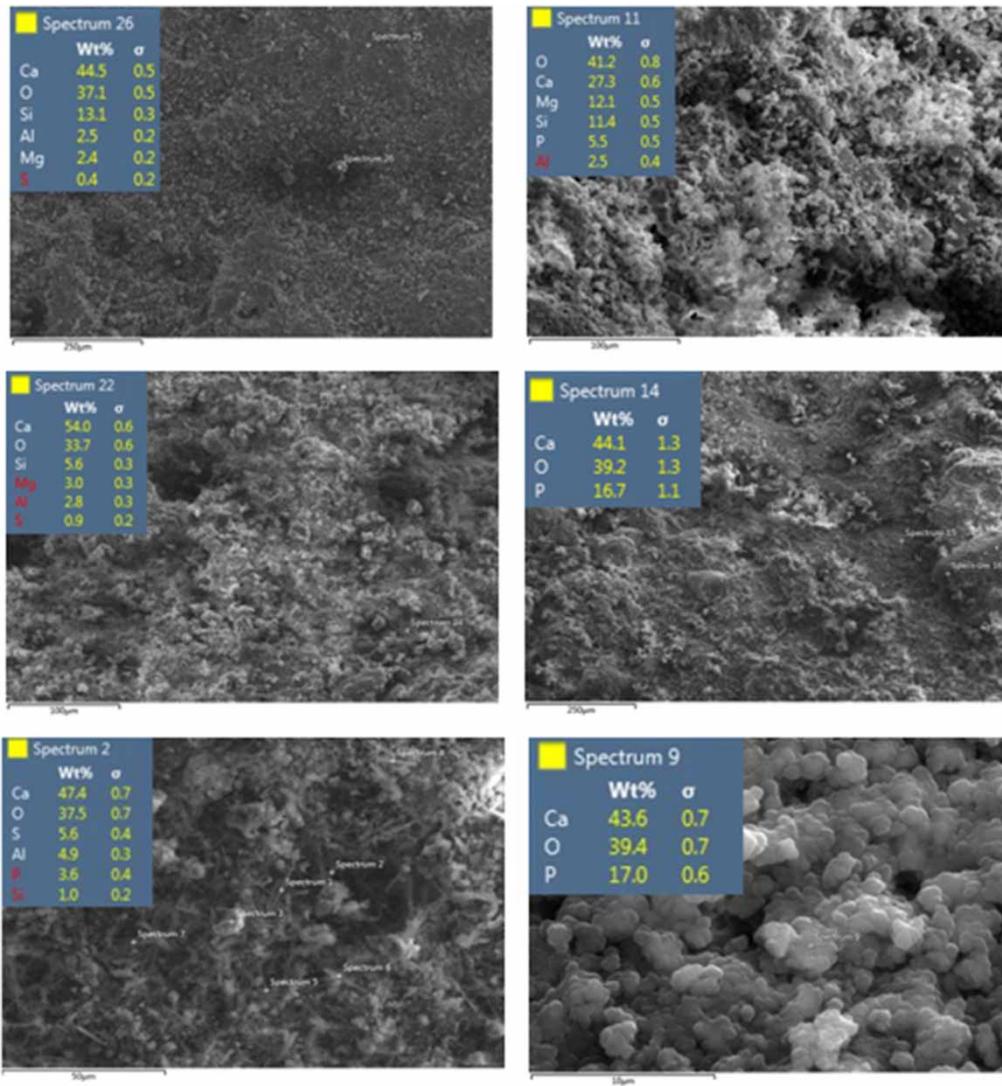


Figure 4 | SEM images of the media before (left) and after (right) the column experiment. From top to bottom: Ca-silicate bedrock, steel slag and apatite 1.

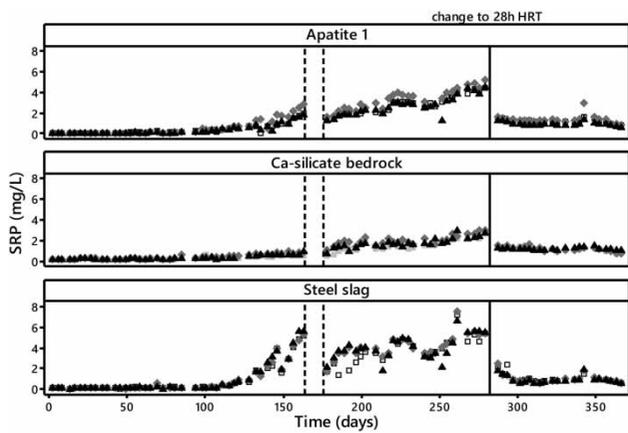


Figure 5 | Media P effluent concentration for the triplicates over the period of 1 year. Change in HRT from 14 to 28 h at day 282 of the experiment, dotted lines indicate the 12-day break.

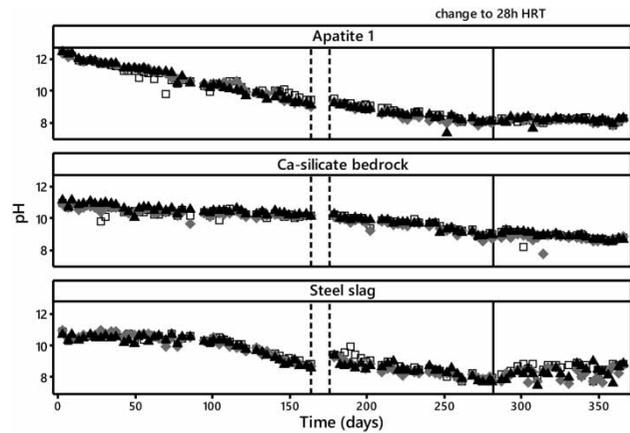


Figure 6 | The pH of the media effluents for the triplicates over the period of 1 year. Change in HRT from 14 to 28 h at day 282 of the experiment, dotted lines indicate the 12-day break.

Table 3 | Mean water quality parameters of column effluents and inlet for 1 year

	Sample <i>n</i>	Inlet	Ca-silicate bedrock	Steel slag	Apatite 1
P removal (%)	94	12.9 ± 4.2 mg/L	93.2 ± 6.2	85.5 ± 18.9	90.3 ± 10.8
pH (min–max)	94	7.2–8.8	7.7–11.1	7.4–11.0	7.2–12.4
BOD ₅ (mg/L DO)	48	2 ± 1.7	1.2 ± 1.0	1.7 ± 1.8	1.3 ± 1.3
COD (mg/L)	19	50 ± 16	39 ± 8	46 ± 15	48 ± 13
TSS (mg/L)	47	6.5 ± 8.7	5.7 ± 16	6.2 ± 12.1	11.9 ± 28.5
TP (%)	14	14.6 ± 5.5 mg/L	88.3 ± 10.7	75.5 ± 21.9	84.8 ± 16.1
Alk. (mg/L CaCO ₃)	28	122 ± 23	145 ± 30	129 ± 45	139 ± 100

Media capacity in mg PO₄-P/g media was calculated via following equation:

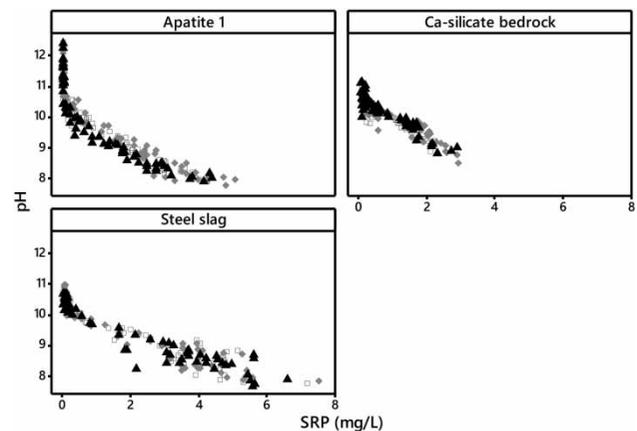
$$Q_t \left(\frac{\text{mg}}{\text{g}} \right) = \frac{(C_0 - C_t) \cdot V}{M} \quad (3)$$

where Q_t is the capacity of P onto the media at time t , c_0 is the inlet P concentration in mg/L and c_t the concentration of P at time t in mg/L, V is the volume of treated wastewater in L and M the mass of media in g.

After 1 year of operation, the columns removed a total of 7.21 mg (Ca-silicate bedrock), 3.28 mg (steel slag) and 3.85 mg of PO₄-P/g media (apatite), which relates to 838, 1,229 and 1,240 pore volumes of treated wastewater, respectively, or a total of 1,196 ± 12 L per column. The Ca-silicate bedrock showed the highest P removal, when the capacity is expressed as mg/g but only because it has the lowest density. This is why a comparison of total mg removed per volume of media is more appropriate for the comparison of the performance between different media. Since the volume of the media was equal in all of the columns (2,356 cm³), the amount of P removed (g) can be compared directly. In total, Ca-silicate bedrock removed the highest amount of P (13.8 g) per column, 12.8 g for steel slag and 13.5 g for apatite.

Relationship between P and pH

As seen in Figure 7, the pH is negatively correlated to the effluent P concentration of the columns. At the beginning of the experiment, when the effluent pH is high, very low P concentrations were observed. Over time, a decrease in P removal was seen along with a drop in pH levels.

**Figure 7** | Correlation between effluent SRP and pH of the columns in triplicates for the first 281 days.

CONCLUSION

The media in this study all showed high P removal efficiencies in both column experiments with very low P effluent concentrations of <0.2 mg/L achieved.

HRT plays a crucial role in the design and sizing of full-size filter beds. From this study, it is evident that a HRT below 8 h is not sufficient to remove P and sustain low concentrations below 1 mg/L for a long period of time. Short HRTs also overloaded the uptake rate of media, which would reduce any economic benefits of smaller filters. However, although higher HRTs are recommended to achieve optimal performance in P removal, they were associated with elevated pH and increased metal concentration in the effluents. Higher HRTs also require a larger volume of media for the design of the filter bed, which increases the footprint of the system and its associated cost. The media effluents do not pose a risk to other consented water quality parameters (such as BOD, COD and TSS) and most cases improved general effluent quality.

The experiments compare different media under the same conditions using actual wastewater rather than synthetic influent, and so are closer to real-world conditions. The systematic comparison of different media and HRTs provides robust design criteria for the scale-up to field scale applications. Therefore, this study demonstrates that the reactive media filters are an appropriate technology for P removal on small sites, especially when combined with a passive pH-correcting system.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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