

## Forms of phosphorus bound to reactive material – results from a flow-through experiment with the focus on P reuse

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### ABSTRACT

P-reactive materials (P-RMs) are used for removing phosphorus (P) from wastewater, surface water, agricultural runoff, green roof runoff and other sources, to protect the water environment against eutrophication. The efficiency of P-RMs in various applications has been widely tested, but there is still a need to expand knowledge of the mechanisms and forms in which retained phosphorus is bound. This is particularly important in regard to possible P recovery. The aim of the study was to evaluate forms of phosphorus bound to reactive material loaded with different P-carrying media: a solution prepared in laboratory from  $\text{KH}_2\text{PO}_4$  (C1) and an effluent from a septic tank (C2). P fractions bound in RM that are fed with P solution and septic tank effluent show different patterns. The Ca- and Mg-bound P are dominant in a filter fed with septic tank effluent (C2), while labile inorganic phosphorus is dominant in a filter fed with  $\text{PO}_4\text{-P}$  solution (C1). It can be stated that tests with P solution did not reflect the P removal mechanisms observed in a filter fed with wastewater, and the suitability of the reactive material as a fertilizer should be inferred from its behavior under real conditions of use.

*Keywords:* Phosphorus; Reactive material; Sorption mechanisms; P recovery

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### 1. Introduction

Phosphorus (P) deposits around the world are depleting, so harvesting phosphorus from waste streams, for example, domestic wastewater, agricultural runoff, and other sources, is gaining more and more interest [1,2]. Effluent from on-site wastewater treatment plants is rich in phosphorus, and has a greater negative impact on the environment than previously believed [3]. Both problems can be solved in a sustainable way through the incorporation of phosphorus reactive materials (P-RMs) into on-site wastewater treatment systems [3–5]. P-RMs can remove P in

a targeted manner from polluted water and the P sorption mainly depends on the content of the Ca, Al or Fe fractions [3,6]. Although much information exists on the efficiency of RMs in P removal in various applications, for example, surface water [7–9], wastewater [10–12] or agricultural runoff [13], comparatively less attention has been given to the mechanisms and forms in which retained phosphorus is bound. The P sorption capacity of RMs is usually a result of laboratory-scale static and dynamic tests with the use of potassium dihydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ), dipotassium hydrogen phosphate ( $\text{K}_2\text{HPO}_4$ ) or monosodium phosphate ( $\text{NaH}_2\text{PO}_4$ ) solution, or synthetic wastewater [14,15] as a

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phosphorus carrier. Real wastewater is not often used in laboratory-scale tests as it creates transportation and storage problems. Tests with P solutions are preferred in some cases, as, if they are carried out under similar conditions, they make the P removal efficiency of different RMs comparable. On the other hand, they do not reflect the conditions that are achieved in technical-scale applications [16]. Some discrepancies reported to date are the blocking of sorption sites by biological film or the acceleration/slowing down of the sorption process by the presence of other compounds or biological factors, such as the competitiveness of algae. This study shows that the processes, and therefore the forms of P bound, are different when RM is tested with P solution and real wastewater. It should be considered whether recovery of the retained P is an option.

The aim of this study was to evaluate forms of phosphorus bound to reactive material loaded with (i) solution prepared from potassium dihydrogen phosphate and (ii) effluent from a septic tank sampled from an on-site wastewater treatment plant. A modified Hedley's Sequential Extraction Procedure was used to recognize forms of P, with the focus on plant-available P, which is particularly important in regard to possible P reuse.

## 2. Materials and methods

This study presents P fractions bound to the Polonite® (Ecofiltration Nordic AB) in grains of 2–6 mm previously used as a filter in a column experiment fed with the solution prepared from  $\text{KH}_2\text{PO}_4$  (C1) and wastewater sampled from the outlet of the septic tank (C2). Polonite® is a material of natural origin, processed by heating. The main chemical components of the Polonite® used in this study are:  $\text{SiO}_2$  (52.08%),  $\text{CaO}$  (22.27%),  $\text{Al}_2\text{O}_3$  (5.57%),  $\text{Fe}_2\text{O}_3$  (2.19%) and  $\text{MgO}$  (0.82%). With the apparent  $\text{PO}_4\text{-P}$  sorption capacity of  $40.9 \text{ mg g}^{-1}$  [17], tested Polonite® was classified as P-reactive [18]. The unit sorption of  $\text{PO}_4\text{-P}$  obtained in a flow-through experiment amounted to  $0.09 \text{ mg g}^{-1}$  for C1 (Polonite® fed with  $\text{PO}_4\text{-P}$  solution) and  $0.14 \text{ mg g}^{-1}$  for C2 (Polonite® fed with wastewater). Wastewater used as a P carrier in C2 was collected from an on-site septic system (6 PE), from a well located between the septic tank and the infiltration system. The wastewater retention time in the septic tank was 3 days and the 5-day biochemical oxygen demand ranged between 400 and 440  $\text{mg L}^{-1}$ , EC 3.0–3.2  $\text{mS cm}^{-1}$  and  $\text{PO}_4\text{-P}$  11.5–18.6  $\text{mg L}^{-1}$ . The wastewater was filtered through paper filters before discharge to filter. As a P carrier for C1, a solution with a  $\text{PO}_4\text{-P}$  concentration of  $10.69 \text{ mg L}^{-1}$  was used. The details of the column experiment and the  $\text{PO}_4\text{-P}$  removal efficiency have been described in [17].

Ten samples of P-containing filtration material were sampled from each column, and were subjected to sequential extraction. The speciation of phosphorus was realized using a modified Hedley's Sequential Extraction Procedure [19]. Samples (1 g of dry weight) and a substrate to solution ratio of 1:30 on a weight (g) to volume (mL) basis were used. The sequential extractants were: (i)  $\text{NH}_4\text{Cl}$  (1 M), (ii)  $\text{NaHCO}_3$  (0.5 M), (iii)  $\text{NaOH}$  (0.1 M) and (iv)  $\text{HCl}$  (1 M). Pools of P identified by the fractionation scheme used in this study included: (i) soluble P, (ii) loosely adsorbed P, (iii) Fe- and Al-bound P, and (iv) Ca-bound P. Phosphorus

extractable by  $\text{NH}_4\text{Cl}$  is called soluble P and is an easily available form of P [20]. Labile inorganic P extracted with 0.5 M  $\text{NaHCO}_3$  represents loosely adsorbed P. It can be readily released into the water column [21]. P extracted using this method was found to be effective in determining plant-available P [20]. The  $\text{NaOH}$ -extracted solution represents phosphorus bound with aluminum and non-reducible iron compounds [22,23]. It represents: (1) P associated with hydroxide surfaces which can be desorbed at elevated pH levels; (2) P associated with crystalline Fe and Al oxides which is only desorbable into solution under extended waterlogged conditions; and (3) P associated with organic matter in humic and fulvic acids (as the difference between the total P of the  $\text{NaOH}$  extract and the soluble reactive P concentration in the  $\text{NaOH}$  extracts) [20]. The fraction extracted with  $\text{HCl}$  represents Ca- and Mg-bound P. Ca and Mg tend to release P under low pH conditions [20]. Calcium phosphate can be used as slow-release fertilizer or as feedstock for regular fertilizer production [1]. At mid to high pH ranges (6.0–8.5), Ca compounds can precipitate [24].

All solutions collected after 24 h of extraction were analyzed for  $\text{PO}_4\text{-P}$  via the ammonium molybdate method in the range of 0.005–1 or 1–5  $\text{mg L}^{-1}$  using a FIAstar Analyzer. The data were evaluated with the Statgraphics Centurion XVI software. A one-way ANOVA (analysis of variance) was used to test the differences between P fractions and when the differences were detected, Tukey post hoc tests were performed in order to determine the nature and magnitude of these differences at the significance level of 0.05.

## 3. Results and discussion

### 3.1. Forms of P identified aligning vertical distribution of P fractions in the filters

The shares of phosphorus fractions in filtration materials fed with P solution and septic tank effluent show different patterns (Fig. 1).

The Ca- and Mg-bound P is dominant (70%) in the filter fed with septic tank effluent (C2). This means that filtration of the mechanically treated wastewater promoted the formation of not easily soluble forms of phosphorus. Ca phosphates are not easily plant-available at pH values  $> 7$ , where hardly plant-available hydroxyapatite will occasionally be formed, which in the case of some RMs can be a very quick process in contrast to hydroxyapatite formation in natural soils [25]. The second important fraction in C2 (22%) is an inorganic P extracted with 0.5 M  $\text{NaHCO}_3$ , which represents loosely adsorbed P. This fraction can be readily released into the water column [20]. In the filter fed with the solution prepared from  $\text{KH}_2\text{PO}_4$  and the tap water (C1), 75% of phosphorus was found in labile inorganic form and only 4% in Ca- and Mg-bound forms. The  $\text{NaOH}$ -P fraction represents about 10% of the forms of P in C1 and 4% of P in filter C2. The content of phosphorus soluble in ammonium chloride ( $\text{NH}_4\text{Cl}$ ) amounted to around 11% (C1) and 4% (C2) of the total content of phosphorus detected in the filtration materials. Fig. 2 shows the distribution of the fractions with the depth of the filters. No trends connected with the share of the P fraction and length of the column were detected in either of the filters.

3.2. Effect of P-carrying media

An ANOVA analysis was performed to analyze the differences between the share of different forms of P in the total amount of P detected in filtration materials sampled from columns C1 and C2 (Table 1). In the case of all P fractions, the ANOVA shows statistically significant differences at the 95% confidence level. Phosphorus bound to calcium was a dominant fraction within the C2, while labile inorganic P extracted with 0.5 M NaHCO<sub>3</sub> was dominant in C1. This suggests that the PO<sub>4</sub>-P supplied in the

form of solution promotes the binding of phosphorus in the plant-available form. A similar observation was made by Johansson and Hylander for the RM of the same origin [26]. The difference in the behavior of the P sorption process in the case of different carrying media suggests that further reuse of bound P can be limited due to the application of reactive material. As the carrying media may have a significant effect on P sorption mechanisms, it may be difficult or even impossible to relate the results of standard laboratory RM tests to the results of full-scale applications.

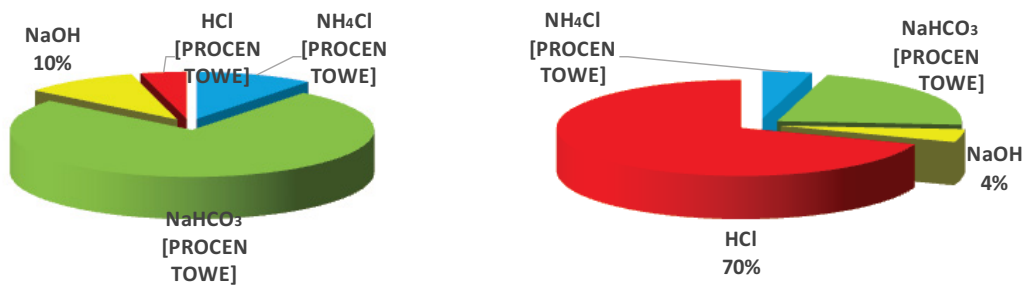


Fig. 1. Phosphorus fractions in filtration material fed with P solution (C1, left) and septic tank effluent (C2, right).

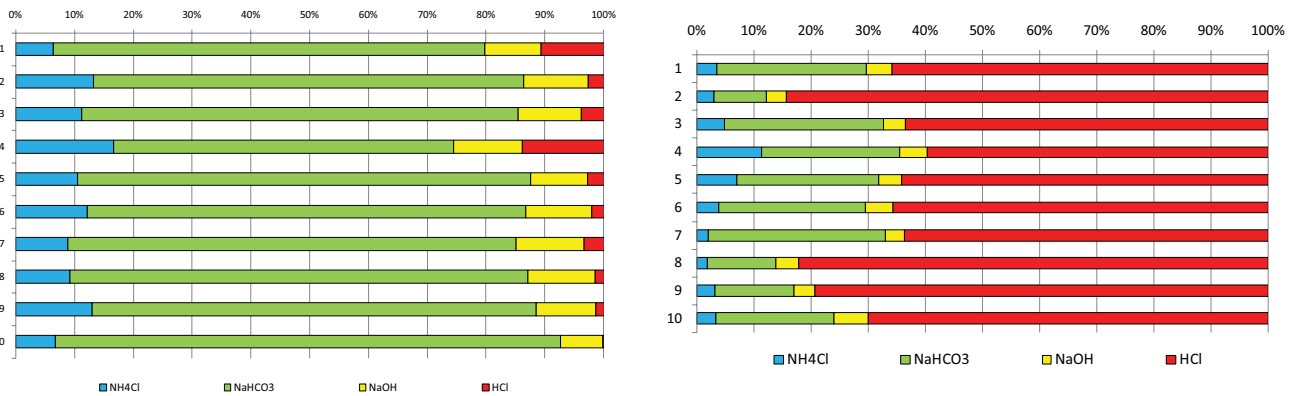


Fig. 2. Vertical distribution of P fractions in RM fed with KH<sub>2</sub>PO<sub>4</sub> solution (C1, left) and septic tank effluent (C2, right). Numbers on the y-axis show the number of the sample from 1 – top to 10 – bottom of the column.

Table 1

Results of the ANOVA analysis showing differences between the shares of different forms of P in the total amount of P detected in filtration materials sampled from columns C1 and C2

| P fraction   | n  | Minimum | Maximum | Average | Median | Geometric mean | Standard deviation | Homogenous groups |
|--|----|---------|---------|---------|--------|----------------|--------------------|-------------------|
| C1 – RM fed with potassium dihydrogen phosphate solution |    |         |         |         |        |                |                    |                   |
| NH <sub>4</sub> Cl                                       | 10 | 0.006   | 0.012   | 0.009   | 0.009  | 0.009          | 0.002              | A                 |
| NaHCO <sub>3</sub>                                       | 10 | 0.031   | 0.079   | 0.058   | 0.056  | 0.056          | 0.013              | C                 |
| NaOH   | 10 | 0.006   | 0.009   | 0.008   | 0.008  | 0.008          | 0.001              | B                 |
| HCl  | 10 | 0.001   | 0.010   | 0.003   | 0.002  | 0.002          | 0.003              | B                 |
| C2 – RM fed with septic tank effluent                    |    |         |         |         |        |                |                    |                   |
| NH <sub>4</sub> Cl                                       | 10 | 0.016   | 0.014   | 0.016   | 0.014  | 0.014          | 0.010              | X                 |
| NaHCO <sub>3</sub>                                       | 10 | 0.079   | 0.087   | 0.079   | 0.087  | 0.072          | 0.032              | Y                 |
| NaOH   | 10 | 0.016   | 0.015   | 0.016   | 0.015  | 0.015          | 0.008              | X                 |
| HCl  | 10 | 0.253   | 0.228   | 0.253   | 0.228  | 0.246          | 0.068              | Z                 |

Table 2

The  $\text{PO}_4\text{-P}$  concentrations, total loads in solution/wastewater applied to filters and detected in leachates, reduction of  $\text{PO}_4\text{-P}$  and unit P sorption on the filters [17], modified by adding a column with extraction results

| $C_{\text{in}}$ (mg L <sup>-1</sup> ) mean<br>(min–max)  | $C_{\text{out}}$ (mg L <sup>-1</sup> ) mean<br>(min–max) | $L_{\text{in}}$ (mg) | $L_{\text{out}}$ (mg) | R (%) | Unit sorption<br>$L_{\text{in}}-L_{\text{out}}$ (mg g <sup>-1</sup> ) | Extracted P (mg g <sup>-1</sup> )<br>(this study) |
|--|--|----------------------|-----------------------|-------|---|---|
| C1 – RM fed with potassium dihydrogen phosphate solution |  |                      |                       |       |   |   |
| 4.41 (0.29–16.75)  | 1.21 (0–4.85)  | 33.44                | 9.21                  | 72.5  | 0.09  | 0.08 ± 0.01                                       |
| C2 – RM fed with septic tank effluent                    |  |                      |                       |       |   |   |
| 10.69 (2.22–20.87)                                       | 4.85 (0.85–13.90)  | 72.83                | 34.39                 | 52.8  | 0.14  | 0.36 ± 0.09                                       |

### 3.3. P forms vs P removal

The total amount of phosphorus extracted from RM fed with potassium dihydrogen phosphate solution reflects the calculated phosphorus removed by the filter during the column experiment [17]. The estimated phosphorus sorption in this filter, calculated based on the difference between the load of  $\text{PO}_4\text{-P}$  in influent and effluent, was 0.09 mg g<sup>-1</sup>, while the extracted was 0.08 ± 0.01 mg g<sup>-1</sup>. In the case of the RM fed with septic tank effluent, the calculated sorption was 0.14 mg g<sup>-1</sup>, while the total extracted P was higher and amounted to 0.36 ± 0.09 mg g<sup>-1</sup>. The amount of loosely adsorbed P was 0.06 ± 0.01 mg g<sup>-1</sup> and 0.08 ± 0.03 mg g<sup>-1</sup> for C1 and C2 respectively. It can be roughly stated that the P introduced in a dissolved form was retained in readily available forms. The surplus of P extracted from C2 in relation to that introduced in the form of  $\text{PO}_4\text{-P}$  may result from the phosphorus in an organic form in the septic tank effluent. Data from the literature indicate that the share of soluble reactive phosphorus in the septic tank effluent ranges between 36% to 77% of total P [27,28]. An average of 0.25 ± 0.07 mg g<sup>-1</sup> was found as the HCl-bound fraction in filter C2. This may suggest that the organic phosphorus present in wastewater tends to create Ca and Mg compounds. However, this can only be hypothesized, not stated, because the column experiment was performed with the focus on the difference between the removal of dissolved P by RMs fed with solution and wastewater and the total P was not analyzed in the septic tank effluent.

### 3.4. Possible P recovery

Taking into account the constant demand for phosphorus and the lack of synthetic substitutes, P removal from wastewater by reactive materials suggests the possibility of using bound phosphorus as a fertilizer [29]. It is believed that reactive materials with a Ca/Mg content have an ability to bind some P in the forms accessible for plants [30]. The idea of nutrient recycling from polluted water is not new, but is becoming more relevant due to the depletion of natural resources, as well as geopolitical problems concerning their availability. Phosphate rock and phosphorus are included on the list of critical raw materials [31]. Wastewater treatment with P-RMs and recycling P from P-saturated materials fit the concept of a circular economy. Some early studies showed that RM-bound P can be used by plants directly [32], and plant yields are in agreement

with extractable P [33]. In our study, plant-available phosphorus (extracted with 0.5 M  $\text{NaHCO}_3$ ) was detected in the case of both RMs fed with wastewater and artificial P solution. The difference between the percentages of P bound in this form was significant and depended on the type of P-carrying media (Fig. 1), but the amount of this P fraction in both filters was similar, 0.06 ± 0.01 mg g<sup>-1</sup> and 0.08 ± 0.03 mg g<sup>-1</sup> for C1 and C2 respectively.

On a technical scale, RMs have great potential for implementation in the treatment of wastewater in small wastewater treatment installations [3] and surface waters polluted by different sources, for example, agricultural runoff or urban runoff. They can also be used in some installations of minor importance [34]. As P-carrying water properties can differ significantly, for example, rainwater, surface water or wastewater with a wide variety of contaminants, the material from this specific application should be evaluated for the prediction of the potential use of P-saturated material in fertilizer. This study only indicates that we can encounter a great difference between the results of laboratory tests and technical-scale applications. Not only the form of P bound, but also the form of RM application will limit the use of the fertilization potential of P-saturated RM. One example is the use of RM as an integral element of technical construction, for example, a drainage layer or substrate component of green roof or a permeable reactive barrier to treat agricultural runoff. But in some other applications, for example, separate filters (column or chamber), suspended reactive filters, bags filters, etc., enable the replacement of P-saturated RM. Those applications are preferable in regard to a circular economy.

## 4. Conclusions

The aim of this study was to evaluate forms of phosphorus bound to reactive material loaded with solutions prepared from potassium dihydrogen phosphate and effluent from the septic tank of on-site septic system. Forms of retained P were recognized with the focus on plant availability, and P that could potentially be easily reused by applying RM as a fertilizer. Based on the results, it can be stated that P-carrying media have significant effects on the forms in which P is bound. Thus, the fertilizing suitability of the P-saturated RM can be assessed based on real study only. The results presented in this publication are part of ongoing research, and the conclusions drawn should be treated as preliminary, requiring confirmation in larger

studies of this type. Further research should be performed to confirm whether there is a correlation between  $\text{PO}_4\text{-P}$  in the treated water/wastewater and available and loosely bound phosphorus. In addition, further research is needed to confirm the hypothesis that the presence of organic phosphorus tends to create Ca and Mg compounds.

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