

Original Research

Fertilizer Potential of Calcium-Rich Substrates Used for Phosphorus Removal from Wastewater

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Abstract

Phosphorus (P) in wastewater is an important source of pollution, but properly managed, it can become a resource. Reactive filter media with a high affinity for P are promising in reducing P from effluents allowing nutrient recycling. In this study, three calcium-rich substrates (Filtru P, Polonite, wollastonite) with ability to remove P from wastewater have been saturated with P and tested as potential fertilizers in a pot experiment. Polonite had a relatively higher P content than Filtru P and wollastonite after saturation. All three materials tended to improve the yield of barley compared with the control treatment. Polonite induced the highest yield per unit of amendment from all three materials due to its higher P content, which could be shown in a higher ammonium lactate (AL)-extractable P in soil after harvesting. The application of the substrates slightly increased soil pH and decreased the hydrolytic acidity.

Keywords: Barley, Filtru P, Polonite, Pot experiment, Wollastonite

Introduction

Phosphorus (P) is an essential macronutrient for crop production, and, in fact, agriculture accounts for about 80% of the phosphate ore utilisation worldwide. But P is a finite and non-renewable resource, and current phosphate deposits may last for only a hundred years at the present depletion rate [1, 2]. Despite diminishing P resource deposits, P concentrations in wastewater and agricultural run-off usually exceed critical values causing eutrophication of surface waters. Properly managed, however, P in wastewater becomes a resource rather than a pollutant. There are currently several alternative ways to remove P from wastewater enabling its future use as fertilizer [3]. Conventional wastewater treatment using chemical precipitation or bio-

logical removal of P generates large amounts of sludge, which is costly to manage and does not always allow efficient P recovery. Advanced onsite wastewater treatment systems using reactive filter media instead of sand or gravel, represent a viable alternative to conventional treatment [4-7]. Such media may consist of a porous material rich in Ca, Fe or Al with a high affinity for P. The mechanisms of P retention involve sorption processes at the surface of the material. There is a large variety of reactive substrates described in literature [4, 7-12].

Once saturated with P the material could be recycled back to agriculture if the content of toxic compounds and pathogens does not restrict its use. P in the material should be in a form capable of desorbing and being released to the soil P solution, thus becoming available to plants. The concentration of P in the soil solution is usually quite low; in fact, more than 80% of soil P becomes immobile and unavailable for

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plant uptake because of adsorption, precipitation and conversion to organic form [13]. Both the P status of the soil and the amount and form of P in the amendment influence the contribution of P from the soil solution to total plant uptake [14]. There are several methods to estimate the plant available soil P that can also be used for P-enriched substrates. The ammonium lactate (AL)-extractable P in acetic acid is one of the most common methods [15]. Water- and CaCl_2 -extractable P are also used. However, chemical extractants do not always indicate the P status satisfactorily [16]. A direct method to assess the efficiency of P-enriched substrates as a fertilizer is the determination of crop response and crop P-uptake in cultivation experiments. A number of pot experiments have recently been conducted in order to study the plant availability of P from P-enriched soils [17], sewage sludge [18, 19] and different substrates used for wastewater treatment [20-22]. In most cases, P-saturated substrates improved the yield compared with no P addition. It has been shown that P bound to Ca compounds is more plant available than P bound to Al and Fe in the studied substrates [20]. It is, therefore, of great interest to evaluate the fertilizer efficiency of calcium-rich substrates saturated with P. In addition, such media give high pH values in the effluents, which effectively reduces the content of bacteria [23].

The objective of this study was to estimate the fertilizer potential of three calcium-rich materials (Filtra P, Polonite, wollastonite) used for wastewater treatment, on the yield and composition of barley and to evaluate the effect of such amendments on soil pH, hydrolytic acidity and availability of P, K and Mg after harvest.

Materials and Methods

Reactive Filter Media

Three different calcium-rich reactive media known for their ability to remove P from wastewater have been used. Polonite with a particle size of 2-5.6 mm, is the product of processing opoka, a bedrock rich in CaCO_3 and SiO_2 that was formed from marine sediments [10]. Filtra P, a product developed by the company Nordkalk, consists of spherical aggregates (2-10 mm) of lime, Fe compounds and gypsum. Wollastonite is a calcium metasilicate compound with reported P sorption ability [8]. Wollastonite tailings are manufactured having a diameter of 1-3 mm. An amount of 200 g of each material was placed in columns made from 1-litre plastic graduated cylinders with perforated base. The columns were gravity fed with a 25 mg P dm^{-3} (as KH_2PO_4) solution until saturation was reached, i.e. the influent and effluent P concentrations did not differ significantly. Then, the materials were removed and equilibrated in a 100 mg P dm^{-3} (as KH_2PO_4) solution for 2 days in order to ensure a homogeneous P content in the substrates. After that, the materials were dried at 105°C and milled for analysis.

The pH of the materials was then measured in a 1:2.5 (w/v) material:water and KCl 1M solution suspension. The total P, Al, Fe, Ca, Mg, Cu, Mn, Cd, Pb and Zn content in the materials was determined by atomic spectrometry using an ICP-AES JY 238 Ultrace after extraction with nitric and perchloric acids by heating for 3-4 days and later filtration.

Pot Experiment

The fertilizer potential of the P-enriched media was tested in a pot experiment using a soil acquired in Łazy, situated 40 km south of Krakow, Poland ($20^\circ30'\text{E}$; $49^\circ58'\text{N}$; altitude 320 m). It is the A horizon (0-25 cm) of a cultivated field, classified as a Haplic Luvisol [24], and consists of 12% sand, 56% silt and 32% clay. The pH, AL-extractable P and K, and total element content (P, Al, Fe, Ca, Mg, Cu, Mn, Cd, Pb and Zn) in the soil are shown in Table 1.

Each pot was filled with 4.0 kg wet soil (30-35 vol.%) and received 1.5 g K_2SO_4 , 0.5 g MgSO_4 and 1.0 g NH_4NO_3 as basic fertilization, incorporated and mixed in the middle-upper soil layer (5-10 cm). P was added as KH_2PO_4 or as P sorbed to the materials in quantities based on previous research [22]. The materials were crushed and sieved to a fraction of 0.5-1 mm before mixing with the soil, in order to enhance P release to the soil solution. There was no P added to the control sample. Each treatment was in triplicate.

Spring barley, *Hordeum vulgare* c.v. Poldek was sown on 16th of May 2006 in a dose of 20 seeds per pot. No fungicide or any other chemical was used to prevent common diseases in order to check the vulnerability of barley to different treatments. The pots were randomly distributed in a greenhouse situated in Krakow ($19^\circ51'54.43''\text{N}$; $50^\circ00'41.30''\text{E}$), Poland, with an insolation of 440 hours during the experiment. The plants were watered every one or two days keeping an average soil moisture of 30-35 vol.%. The pots were relocated within the greenhouse every one or two weeks. The average air temperature during the experiment in the greenhouse was $15\text{-}20^\circ\text{C}$. Harvesting took place on 18th of July (64 days), when the spikes were already developed and before reaching any critical stage of disease. The plants were cut manually at approximately 1 cm above the soil surface, dried at 55°C and weighed. Next, spikes from barley were cut and milled. The total P, Al, Fe, Ca, Mg, Cu, Mn, Cd, Pb and Zn content was determined by atomic spectrometry using an ICP-AES JY 238 Ultrace after extraction with a mixture of nitric and perchloric acids at 200°C under recirculation conditions.

After harvesting, soil samples from each pot were analysed for AL-extractable P, K and Mg in acetic acid [15] and hydrolytic acidity according to the Kappen method using 1 mol dm^{-3} CH_3COONa solution. The pH was measured in a 1:2.5 (w/v) soil:water and KCl 1M solution suspension.

Table 1. Physical properties, total element concentration, AL-extractable P and K, and pH in the reference soil and the materials after P-saturation

Parameter	Soil	Polonite	Filtra P	Wollastonite
Particle size (mm)	-	2-5.6	2-10	1-3
Bulk density (g cm ⁻³)	-	0.7	1.0	1.4
pH _{H₂O} / pH _{KCl}	6.88 / 6.42	9.88 / 9.90	11.49 / 11.36	9.02 / 8.76
Ca	0.86 ± 0.01	230.44 ± 1.49	343.22 ± 0.69	162.41 ± 4.21
Mg	1.64 ± 0.05	5.01 ± 0.06	5.61 ± 0.04	1.76 ± 0.08
Fe	15.99 ± 0.22	11.15 ± 0.14	44.42 ± 0.37	7.55 ± 0.74
P	663.73 ± 7.85	1862.15 ± 53.58	713.64 ± 115.99	253.96 ± 12.73
Mn	246.43 ± 3.53	49.90 ± 0.31	195.65 ± 1.17	394.87 ± 15.10
Zn	52.86 ± 0.58	53.18 ± 0.33	92.84 ± 1.10	21.34 ± 0.07
Cu	12.26 ± 0.10	4.41 ± 0.04	5.98 ± 0.20	3.12 ± 0.11
Pb	15.49 ± 0.33	0.64 ± 0.17	7.01 ± 0.27	0.97 ± 0.07
Cd	0.16 ± 0.08	< 0.5	< 0.5	< 0.5
AL-P	7.51 ± 0.15	49.88 ± 0.31	5.62 ± 0.38	13.93 ± 0.61
AL-K	61.33 ± 0.24	62.13 ± 0.02	23.48 ± 0.58	4.49 ± 0.01

Results and Discussion

P Content in the Materials

The physical properties, total element concentration (P, Ca, Fe, Mg, Mn, Cu, Zn, Pb, Cd), AL-extractable P and K, and pH in the materials after saturation is shown in Table 1. The P content in Polonite (nearly 2 g P kg⁻¹) was considerably higher than that of Filtra P and wollastonite. Previous work using Polonite for wastewater treatment showed a P content of about 1.3 g P kg⁻¹, suggesting that the material was not fully saturated [22]. The lower P content in Filtra P suggests that precipitated phosphate may have been washed out of the column. Supporting this explanation is the fact that Filtra P disintegrates in water with time giving a yellow colour to the effluent. On the other hand, wollastonite effluents were very clear, but its sorption capacity was much lower. Previous research has shown that wollastonite can remove over 80% P from wastewater when using hydraulic residence times of more than 40 h. [8]. In that study, tailings with pure wollastonite powder were used, thus having a larger surface area.

The method used to saturate the materials may not be adequate to estimate their longevity according to Drizo et al. [9]. However, it was used with the only purpose of saturating the three different materials with P under similar conditions. The expected P accumulation in the materials when filtering wastewater might be relatively lower than that shown in Table 1 because organic matter and other compounds present in wastewater may reduce P sorption and precipitation.

The pH value of Filtra P was still very alkaline after saturation decreasing from 12.5 to 11.5. The pH of Polonite decreased from 12.0 to about 9.9 as a consequence of increasing P saturation. The pH was almost unaffected by P saturation for wollastonite (dropping from 9.4 to 9.0). The element content in the materials after saturation (Table 1) confirmed that all three materials are rich in Ca, but Filtra P has a considerably higher content. Polonite and Filtra P contain substantial amounts of Mg. Filtra P contains also large amounts of Fe. Wollastonite contains considerable amounts of Mn. All three materials have no trace of Cd. The content of Cu is similar in the three materials but Zn differs and it is highest in Filtra P, which also contains considerably higher amounts of Pb than the other two materials. Polonite and wollastonite have a noticeably high content of Si (not shown).

The AL-extractable P in the substrates (Table 1) was quite low compared to other substrates studied earlier [25], and it was especially low for Filtra P. In addition to the limitations of plant availability tests using chemical extractants [16], the estimated AL-extractable P in the substrates is not so representative when using them as soil amendments, in which case, it is the net AL-extractable P from the soil-substrate mixture that matters. The AL-extractable K (Table 1) was particularly high in Polonite (similar to that of the reference soil) and much lower for the other materials.

Fertilizer Potential of P-Saturated Media

The yield of barley grown under five different treatments is shown in Figure 1. There is no significant differ-

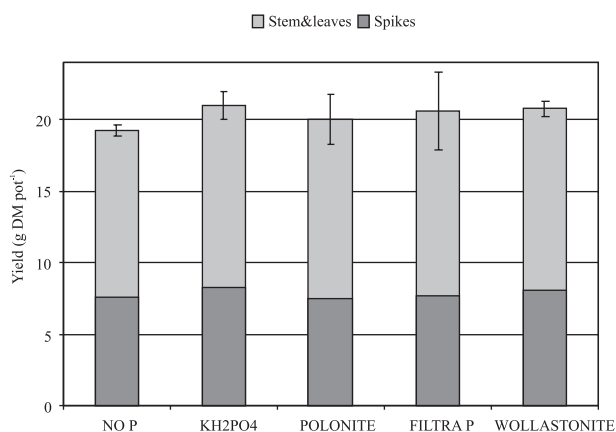


Fig. 1. Average yield of barley grown under different treatments and dry matter distribution (bars indicating SE for total yield).

ence in dry matter (DM) production between treatments but there is a tendency to higher yield when applying P in a water-soluble form (KH_2PO_4) and P from saturated media compared with the control treatment (No P). The addition of 0.12 g of KH_2PO_4 increased the average yield by nearly 2 g DM in comparison with the control treatment. All three materials induced an increase of about 1

g DM compared with the control treatment. This increase may be due to both additional P supply for plant uptake and the effect of other constituents of the material itself such as Ca, Fe, Mg, Mn, Zn, which can influence plant growth. The substrates may directly provide P or enhance soil P availability. There is no evidence of direct P release from the substrates to the soil solution in this study, and it can only be assumed from the AL-extraction test (Table 1). In order to prove direct P supply it is necessary to use ^{32}P labelling in the substrate and measure its concentration in plants after harvesting [14].

In order to compare the effectiveness of the substrates, the yield per unit of amendment was calculated (Table 2). This factor is relative and it is only valid to compare treatments under similar conditions such as type of soil, plant species, water regime and insolation. Polonite was the most effective substrate treatment according to the yield per unit of amendment, probably due to its much higher P content. Filtra P was almost as effective as Polonite, and wollastonite was the least effective substrate treatment although it gave the highest DM production from all three materials in this experiment. Considering the much lower P content in Filtra P and specially in wollastonite, compared with Polonite, and the lower AL-extractable P from wollastonite and particularly from Filtra P, there might be

Table 2. Amendment rate and yield of barley grown under different treatments.

Treatment	Amendment (g pot ⁻¹)	Yield ± SE (g DM pot ⁻¹)	Spike yield ± SE (g DM pot ⁻¹)	Yield per unit amendment
No P	-	19.23 ± 0.33	7.60 ± 0.32	
KH_2PO_4	0.12	21.00 ± 0.98	8.26 ± 1.08	-
Polonite	22	20.03 ± 1.70	7.53 ± 1.09	0.91
Filtra P	24	20.60 ± 2.72	7.76 ± 1.17	0.85
Wollastonite	32	20.73 ± 0.54	8.06 ± 0.69	0.64

Table 3. Element concentration in barley spikes grown under different treatments (average concentration ± SE)

Element		No P	KH_2PO_4	Polonite	Filtra P	Wollastonite
P	(g kg ⁻¹)	4.59 ± 0.24	4.09 ± 0.17	4.16 ± 0.24	4.83 ± 0.16	4.48 ± 0.21
Ca	(mg kg ⁻¹)	112.12 ± 14.83	147.43 ± 14.26	129.96 ± 12.30	117.22 ± 10.71	146.73 ± 21.15
Mg		792.79 ± 68.86	782.24 ± 71.89	736.85 ± 47.28	867.67 ± 45.53	706.78 ± 44.58
Fe		99.77 ± 5.38	94.55 ± 12.90	90.86 ± 5.36	94.89 ± 2.44	76.69 ± 4.95
Al		59.75 ± 2.30	57.58 ± 1.86	51.24 ± 5.00	50.69 ± 4.32	45.93 ± 1.88
Mn		21.35 ± 3.09	31.86 ± 5.68	21.66 ± 1.17	17.63 ± 2.36	23.21 ± 4.32
Zn		49.02 ± 1.98	47.50 ± 2.62	43.48 ± 2.16	45.10 ± 2.33	42.32 ± 1.55
Cu		8.64 ± 1.34	8.84 ± 2.20	8.94 ± 2.38	10.0 ± 2.39	9.86 ± 2.92
Pb		5.22 ± 0.71	7.83 ± 0.82	8.61 ± 1.85	5.82 ± 0.73	10.25 ± 2.11
Cd		0.48 ± 0.12	0.40 ± 0.08	0.39 ± 0.05	0.49 ± 0.06	0.32 ± 0.10

Table 4. Soil pH and AL-extractable P, K and Mg after harvesting.

Treatment	pH _{H₂O}	pH _{KCl}	AL-P ± SE (mg kg ⁻¹)	AL-K ± SE (mg kg ⁻¹)	AL-Mg ± SE (mg kg ⁻¹)
No P	6.77	6.50	11.71 ± 0.26	24.11 ± 1.49	657.19 ± 4.48
KH ₂ PO ₄	6.83	6.38	12.72 ± 0.49	24.35 ± 0.94	634.72 ± 16.14
Polonite	7.34	7.04	13.88 ± 0.10	24.13 ± 0.93	656.94 ± 24.58
FiltraP	7.24	7.17	13.65 ± 0.32	21.15 ± 0.75	709.03 ± 13.24
Wollastonite	7.18	6.79	12.89 ± 0.40	22.60 ± 0.48	651.00 ± 40.16

other reasons to explain yield increase in addition to direct P supply from the substrates. It is also possible that all three substrates were able to release P at a rate corresponding to the requirements of the growing plants during this short-term experiment.

The substrates were added in amounts based on previous research [22] for Polonite and Filtra P and slightly larger amounts for wollastonite, considering its lower total P content after saturation. In contrast to previous work where the P-enriched substrates were added in different quantities according to its P content in order to reach the same total P application, this study compared more similar doses of added material resulting in different P applications. This procedure may be appropriate for assessing the fertilizer potential of substrates, especially if P is not the only limiting nutrient. That study [22] concluded that crystalline slag (CS) had greater fertilizer effectiveness than Polonite when applying 100 g pot⁻¹ of CS compared to only 18-29 g pot⁻¹ of Polonite (in both cases containing a total P of 0.03 g pot⁻¹). However, such a huge difference in the dose makes it difficult to compare the results because CS introduces other essential elements such as K, Fe, Mg or Mn in higher quantities, which, in case those elements are in shortage, may influence plant growth. According to the yield per unit of amendment, Polonite showed a greater effectiveness than CS in that study.

The plant DM distribution was similar for all treatments and the spike mass did not differ significantly (Figure 1). The element concentration in barley spikes revealed no significant difference between treatments (Table 3). These data suggests that substrate treatment had no effect on barley spike composition.

Soil Test After Harvesting

The soil pH was initially 6.88/6.22 (H₂O/KCl) and did not change during the experiment for the control and KH₂PO₄ treatments (Table 4). However, it slightly increased with the substrate treatment since they all had a relatively high pH. Filtra P did not increase soil pH more than the other substrates despite having a more alkaline pH value (Table 1).

The AL-extractable P considerably increased (from 7.5 to 11.71-13.88 mg kg⁻¹) during the experiment for all

treatments, but relatively more for the treatments with substrates. The higher value of AL-extractable P from soils amended with Polonite and Filtra P in comparison with P fertilized soil, suggests that the substrates could provide more P in a long term run. There was a decrease in AL-extractable K in soil under all treatments from an initial 61 mg kg⁻¹ to around 24 mg kg⁻¹ for control, P fertilizer and Polonite treatment and even lower than that (around 21 mg kg⁻¹) with the application of Filtra P. This may be due to insufficient K fertilizing. There is no significant difference in the AL-extractable Mg from soil under different treatments. The hydrolytic acidity decreased when applying all three substrates compared with the control treatment since the content of basic elements in the materials may have substituted acidic ions in the sorption complex thus improving soil productivity. The observed effects of the amendments on soil properties may be different in the long-term and therefore these results must be taken with caution.

Conclusion

Calcium-rich substrates are appropriate for P recycling from wastewater to agriculture since they have a moderate to high P sorption ability and can be used as soil amendments and improve crop yield. Once saturated, the P content in the substrate is an indicator of the fertilizer effectiveness. The P content in Polonite was higher than that of Filtra P and wollastonite after saturation under similar conditions. All three materials tended to improved the yield of barley, when applied to an arable soil with rather low AL-extractable P, compared with the control treatment, but did not improve it as much as KH₂PO₄. The substrates improved the yield either by direct P supply or improving soil P availability. Although wollastonite produced the highest yield in this experiment, Polonite gave the highest dry matter production per unit of amendment, due to a much higher P content. This was confirmed with the observed highest AL-extractable P from soil amended with Polonite. The amendments did neither alter the plant DM distribution nor the element concentration in the spikes. All three materials increased soil pH and decreased the hydrolytic acidity. The substrate treatment did not alter other studied soil properties.

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