

# Phosphorus Recycling from Wastes

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## Abstract

The plant phosphorus (P) uptake by Italian ryegrass using organic wastes such as sewage sludge, biochar, composted sewage sludge and spent wetland filters as fertilizer was investigated in pot experiments after manipulating the wastes pH by mixing with other wastes giving acidification by acetic acid and compost leachate, and liming by concrete waste and lime. Pots with no fertilizer and with mineral fertilizer served as control. Available P was measured both with passive diffuse gradient thin film (DGT) samplers and by P uptake in the grass. The pH in the treated waste was about 4 in the acid treatment, and more than 11 in the high pH treatment. The pH in the pot during the grass production was adjusted to normal levels. The P uptake increased up to 56% after pH treatment of the waste. The P uptake responded both to the high and low pH treatments, probably due to the prevalence of different P species. The more extreme pH treatments gave the highest uptake. The DGT uptake gave the same broad picture in the pots fertilized with biochar but not in pots with sludge, and, also, without the separation between high and low treatments. The passive samplers correlated relatively well with the measured grass uptake. A principal component (PCA) analysis showed that the P uptake as measured by the DGT correlated with P, potassium (K) and silicon (Si) concentrations, at to a lower degree with iron (Fe) and lead (Pb), and was uncorrelated with nickel (Ni) and copper (Cu). In summary we can say that the pH treatment of the different organic wastes increased the plant P availability. The smallest increase was in the wetland filter that also had the lowest P total uptake. Also the lupin treatment increased the plant P uptake.

## Keywords

Organic Waste, Sewage Sludge, Composted Sludge, Biochar, Wetland Filters, Waste Mixing, pH Treatment, Phosphorus Plant Uptake

## 1. Introduction

Phosphorus (P) is an essential and irreplaceable factor in food production. As

the world P resources are limited, the recycling of P in the food web becomes more and more important. The P content in soil is generally too low to sustain agricultural production. This is especially the case in Australia [1]. Soil P can be separated into 5 categories, 1) plant available mineral P as phosphate ions, 2) organic P, 3) adsorbed P, 4) aluminum (Al) and Fe precipitated phosphates and 5) primary mineral P (mainly apatite), the latter three not directly plant available [2]. Unlike nitrate, which readily moves in soil towards the roots via both mass flow and diffusion, phosphate is immobile. Mass flow typically delivers as little as 1% - 5% of a plant's P demand, and the amount intercepted by growing roots is only half of that [3].

Lowering the pH or increasing the Al concentration makes P precipitate as  $\text{AlPO}_4$ , similar to the precipitation of  $\text{Al}(\text{OH})_3$ . In calcium (Ca) rich soil with high pH levels P precipitates as hardly water soluble Ca-compounds such as e.g. hydroxyapatite, fluorapatite and chlorapatite [4] (Wang & Li, 2010). Work in the Nordic countries has shown a relatively high accumulation of P in peat soils that received treated sewage or industrial effluents for many years [5]. The best immobilization happened in peat that had received effluent that had been treated with  $\text{FeCl}_2$  or Al precipitation agents as part of their treatment before discharge. Investigations in Scandinavian wetlands showed a content of P in filters of up to 7500 mg/kg dry matter [6]. The uptake of P in ryegrass pot experiments was, however, low after some years of operation, just 3% of the P applied. In filters saturated with P as sodium phosphate, the average uptake increased to 24%, and 37% when applied as calcium phosphate fertilizer.

The reuse of P from used wetland filter media showed that the P was less available than P from phosphate fertilizer for small plants with limited root growth. For second and third harvest of well-developed grassland, the wetland filters and mineral fertilizers had almost the same fertilization effect [6] [7]. In addition, the wetland filters may have a liming effect, which increases the availability of phosphorus in acidic soils. The adding of simple organic acids to soil reduced P retention [8]. Wetlands becoming anaerobic will release some of its retained P [9], especially if it is adsorbed to Fe. Some plant species such as *Proteaceae*, or lupins, specialize in utilizing phosphorous in P-depleted soils [1]. P in soil transfers to the roots by mass flow and diffusion, the latter being very low compared to other nutrients. There is an urgent need to develop crops that efficiently utilize inorganic soil P (Pi) by using plants that use root exudates [1].

Fe- and Al bound phosphate is more available when the pH in the soil is above 6.5, as is nitrogen (N), potassium(K), sulphur (S) and magnesium(Mg), whereas Fe and mangan (Mn) are more available at lower pH [6].

Diffuse Gradient Thin-films (DGTs) can be used to predict plant response to P [10] [11] [12]. The DGT effective concentration, which is lower than the true pore water concentration, was to be 10% to 50% of the P solute in soil with low and high P-AL [13].

This project looks at reclaiming P from waste from a waste handler's perspective, by mixing different ready available waste fractions; Cantreatment of the

waste materials by changing their pH improve the plant P availability? Is passive sampling a good way to model plant P availability using waste products as fertilizer?

## 2. Methods

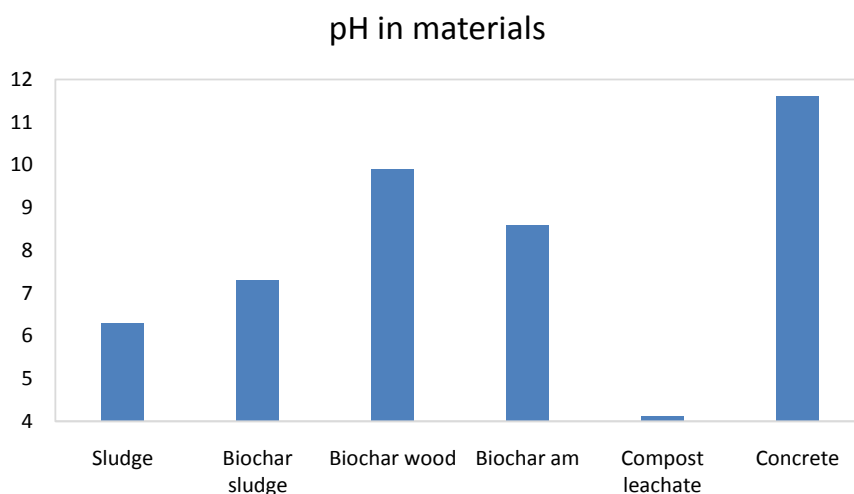
### 2.1. The Materials

Sewage sludge was obtained from Nordre Folloresseanlegg in Ski, Norway. The plant includes a secondary treatment with chemical flocculation and nitrogen removal, using Fe and Al based coagulants in addition to polymers. The sludge used in the experiment had been anaerobically stabilized and stored outdoors for several months (**Figure 1**).

The pH obtained during treatment is shown in **Figure 2**. The pH in the sludge and biochar varies between 6.3 and 9.9, the highest in the biochar produced from wood. Compost leachate is acidic with a pH of close to 4, and concrete waste is very basic with a pH of 11.5. Treatments with low pH received compost



**Figure 1.** Sewage sludge.



**Figure 2.** pH in the waste materials.

leachate at low and high dosage. Treatments with high pH received concrete dust in a similar way. The pH in the low pH treatment was 4.8 and 4.7 with high and low dosage, respectively, and 5.1 and 4.9 in the biochar, and the high pH treatment pH was 9.9 and 11.1 for the sludge, and 11.9 and 11.5 for the biochar. High level included a mix of waste and biochar/concrete at a ratio of 1:2, while low level at ratio 1:1.

The biochar is from Sonnenerde and consisted of two types, mixed together on a 50/50 weight basis (Figure 3). The P rich biochar was produced from sewage sludge, the low P biochar was made from wood.

Figures 4-9 and Table 1 characterize the waste materials. The sludge has some total-P but is low in phosphate probably because they are mainly Al and Fe precipitates, the biochar from sludge is rich in P, most of it as phosphate (Figure 4). The concrete waste does not contain any P. Biochar made from sludge has the highest content of total-P, followed by sewage sludge (Figure 4). Composted



Figure 3. Biochar.

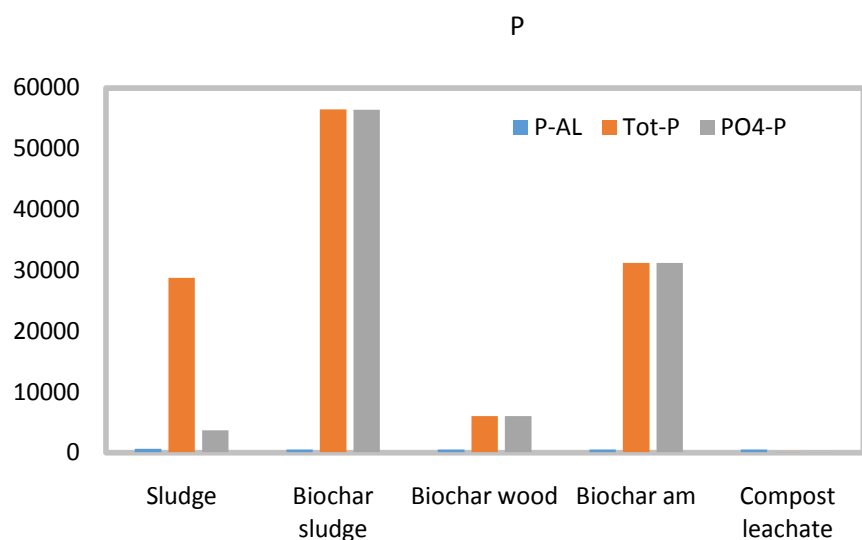
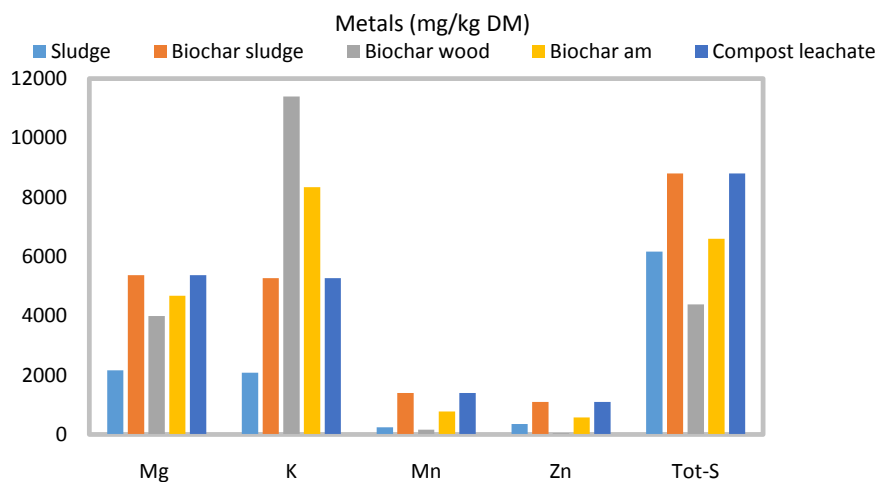
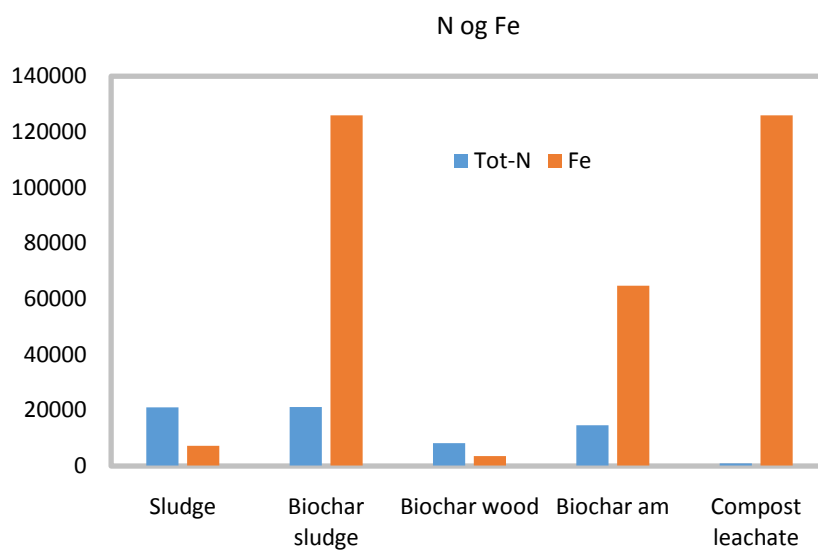


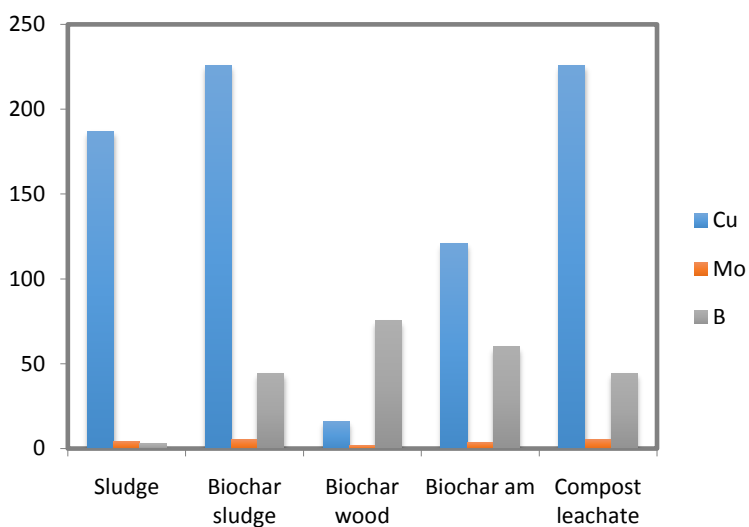
Figure 4. P in the waste materials.



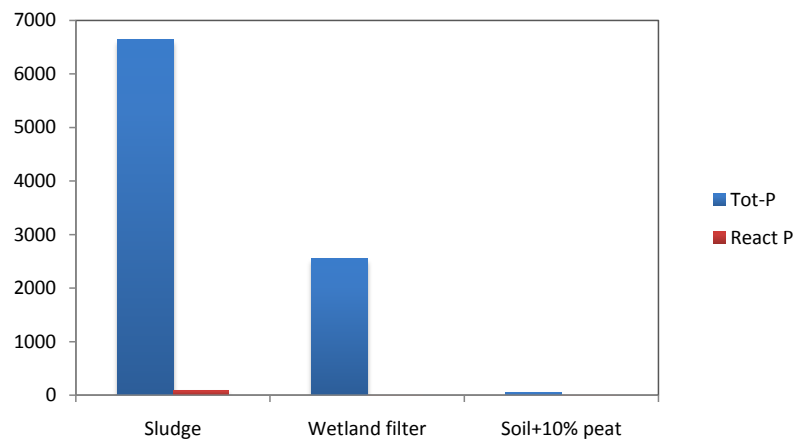
**Figure 5.** Metals and Sulphur in the waste materials.



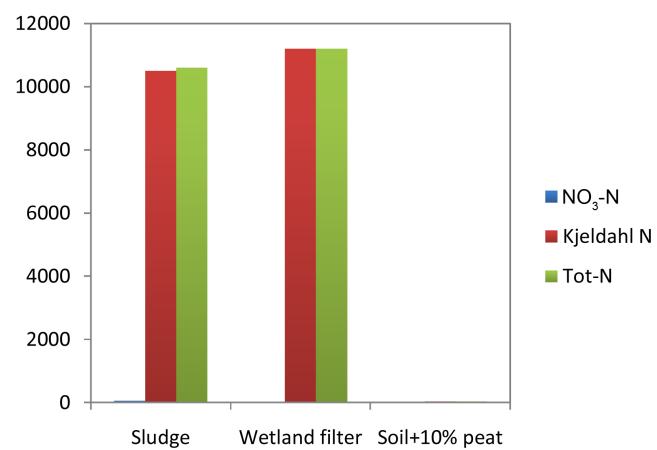
**Figure 6.** N and Fe in the waste materials.



**Figure 7.** Minor nutrients in the waste materials.



**Figure 8.** P in the composted sludge and wetland filter (mg/kg DM).



**Figure 9.** N in the composted sludge and wetland filter (mg/kg DM).

**Table 1.** Characterization of the waste materials and soil/peat mix (in mg/kg dry matter except TOC in %).

	Composted Sludge	Wetland filter	Soil+10% peat
Tot-P	6650	2560	56
React P	94.8	1	<0.1
NO <sub>3</sub> -N	59.3	0.5	<0.1
Kjeldahl-N	10,500	11,200	40
Tot-N	10,600	11,200	40
SO <sub>4</sub>	1550	160	20
Ca	16,900	1410	130
Mg	3870	950	<50
K	6440	950	130
Fe	24,000	26,600	1400
Mg	312	968	<5
Zinc	254	81	<5
TOC	13.2	8	0.25
C/N	12	7	63
Fe/P	3.6	10.4	25



sludge has a content of total-P ten times lower than biochar (**Table 1**). Biochar made from wood is high in potassium (**Figure 5**).

The second part of the experiment used commercial sewage sludge compost and wetland filter as fertilizer (**Table 1**). Both materials contain a lot of P but the amount of reactive P is low. The fertilization was the same as in the first experiment.

The ratio between iron and phosphorus is said to be important for plant P availability and should not be too high. Plant P uptake was significantly higher when the Fe/P ratio was 1.6 compared to 9.8 according to [14]. The sewage sludge had a favorable ratio of 0.3, the biochar 2.2 and the other materials more unfavorable with ratios at 3.6 and 10.4 (**Table 1**).

## 2.2. The Experiments

The experiments were carried out with Italian ryegrass (*Lolium multiflorum*) in 3 liter plastic pots (**Figure 10**) filled with sand with 10% peat as a source for organic matter, while the DGT pots were 1 liter and filled with identical materials. The pots were fertilized with 60 g phosphate and 300 g N, except for unfertilized control pots. N and K were also supplied at the two intermediate harvests, but at a lower rate. The water content of the pots was kept at maximum 90% of field capacity by hand irrigation over 76 days. The harvests from the pots with sewage sludge and biochar were done on day 27, 41 and 76, and from the pots with composted sewage sludge and wetland filters on days 23 and 58.

The field capacity of the pots was estimated by draining the pot under gravity. All measurement had three replicates. The first experiment consists of the following treatments: blank (soil + 10% peat), fertilized with sewage sludge (S), fertilized with biochar (B), fertilized with biochar or sludge that was previously amended with concrete (K) at low or high concentration (high pH treatment with low or high intensity) (BKH, BKL, SKH, SKL), and finally fertilized with biochar or sludge and amended with compost leachate (C) at low or high concentration (low pH treatment with low or high intensity) (BCH, BCL, SCH, SCL).



**Figure 10.** Grass pots.

The experiment using sludge compost and wetland filter consists of the following treatments: blank (soil + 10% peat), fertilized with commercial composted sludge (S), fertilized with wetland filter material (W), fertilized with sludge or wetland filter that was previously amended with lime (L) at low or high concentration (high pH treatment with low or high intensity) (SV or SL), and finally fertilized with sludge or wetland filter and amended with vinegar (V) at low or high concentration (low pH treatment with low or high intensity) (WV or WL).

DGT passive samplers labelled “Loaded DGT device for As and P” are from [15]. The DGTs were kept cold until used and applied in one-liter plastic pots after the material was kept under water saturation for 47 hours on the average, and at a temperature of 15 degrees C. The device was inserted in the pot after making a depression in the soil-waste mixture.

The moisture content in the pots was between field capacity (FC) and 70% of FC by manually refilling the pots.

pH in the materials is determined by mixing with  $2 \times$  volume distilled water overnight.

Each pot received 300 mg Italian rye grass. The pots were additionally fertilized with 300 mg N (as  $\text{Ca}(\text{NO}_3)_2$ ) and K (as  $\text{K}_2\text{SO}_4$ ) in the start of the experiment, and an additional 100 mg after each harvest.

The lupin treatments received 6 seeds, 3 of each species, mandelup and luxor.

Loaded DGT devices for As and P were obtained from DGT Research LTD. The materials were soaked in distilled water for 48 hours before installing the DGTs, and the DGTs stayed in the materials for another 48 hrs, before added to 65%  $\text{HNO}_3$ , and subsequently diluted to 10% before analyzed directly by GC-MS. The results were corrected for temperature dependent diffusion according to the manual given for the samplers (DGT Research, undated).

## 2.3. Analyses

### 2.3.1. Plants

Carbon, hydrogen and nitrogen in the plant material was analyzed in an elemental Vario EL KAN20000 with a TCD detector (Ogner *et al.*, 2000). The sample (5 - 50 mg) is weighed into a tin foil boat. The boat is closed and compressed before introduced into the instrument, and combusted at 950 C. The resulting gases are reduced on a copper column. C and H are adsorbed on adsorption columns, N measures directly. After desorption C and H are measured. Dry matter was measured in a TOR0000 drying cabinet at 105 C. The sample was dried until constant weight.

### 2.3.2. Total Elements

Total elements in plants and DGTs were analyzed by a ThermoJarell Ash-ICP-IRIS HR Duo instrument ICP21100. The sample is blended with digestion acid (72%  $\text{HClO}_4$  + 65%  $\text{HNO}_3$  (1 + 5) v/v) in a teflon bomb. The DGT samples were digested in a 3 ml vial with 10%  $\text{HNO}_3$ . The sealed bombs are heated in a microwave oven. The heating is controlled so only negligible amounts of gas escape



through the safety release valve. The bombs are cooled and the sample diluted with water. The elements Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sc, Se, Sr, Ti, V, Y and Zn are determined by a simultaneous ICP-AES technique by axial or radial viewing of plasma.

### 2.3.3. Wetland Filters and Sludge

The waste and leachate samples were analyzed at the ALS laboratory in Norway, for the parameters total nitrogen, total phosphorus, phosphate, AL extractable phosphorus, total sulphur and the metals B, Cu, K, Fe, Mg, Mn, Mo, and Zn, in addition to pH, TOC and dry matter. Leachate total P was measured spectrophotometrically according to method EN ISO 6878: 2004, and total N according to method 11905-1:1998. Sludge and biochar total P and N was measured spectrophotometrically according to methods CSN 72 0116-1 and ISO 11261, respectively. Total P was calculated from  $P_2O_5$ . Metals in waste and leachate were measured by ICP method DS259, and in leachate with method DS204.

## 3. Results

There was a significant higher evapotranspiration under the high pH treatments both for pots with biochar and sludge, for dry matter production, the biochar pots were higher, also the highest for the concrete treatments. The ET was approximately 3 mm/day in pots with vegetation, and 2 mm/day in blank with only soil.

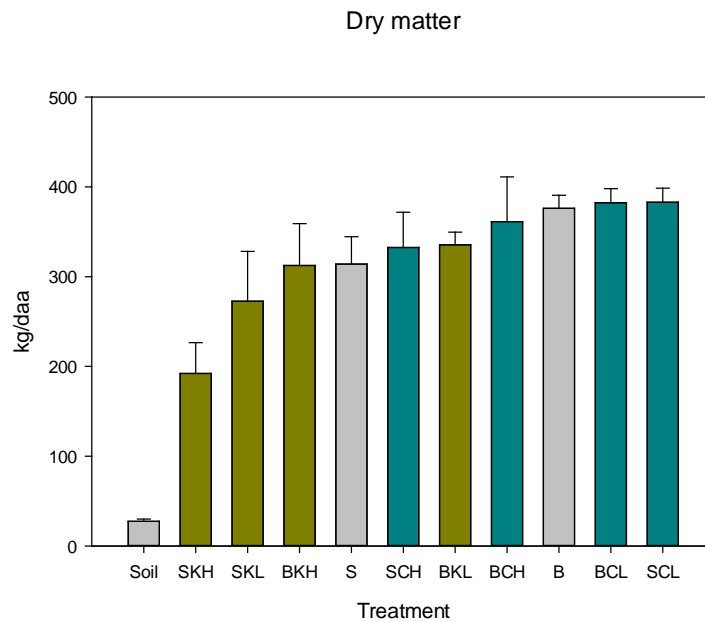
The pots with the lupin treatment showed higher yields, this was obvious from observing the pots (**Figure 11**). The harvest from the pots fertilized with composted sludge and wetland filter (**Figure 12**) was between 25 kg/daa to ca 100 kg/daa, significantly lower than the harvest of the sludge and biochar pots of 150 kg/daa to 300 kg/daa, due to differences in the length of the growing seasons. The harvest was relatively low both in the sludge and wetland pots, compared to the control with mineral fertilizer (**Figure 13**). The sludge pots were higher than the wetland pots. There was a tendency to higher dry matter production in the low pH treatments.

All the pots showed final pH values between 6 and 7, favorable for plant growth. The pH in the DGT pots with concrete treatments was slightly higher.

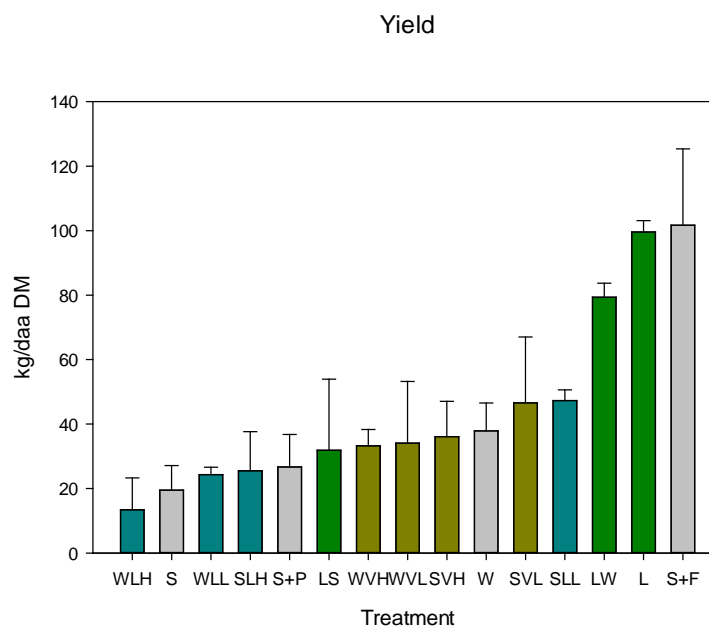
The P uptake was significantly higher in the pots fertilized with biochar (**Figure 14**), with a tendency to higher values in the high pH treatments (BK). In the



**Figure 11.** Pots with lupins before seeded with grass (left) and with grass (right).



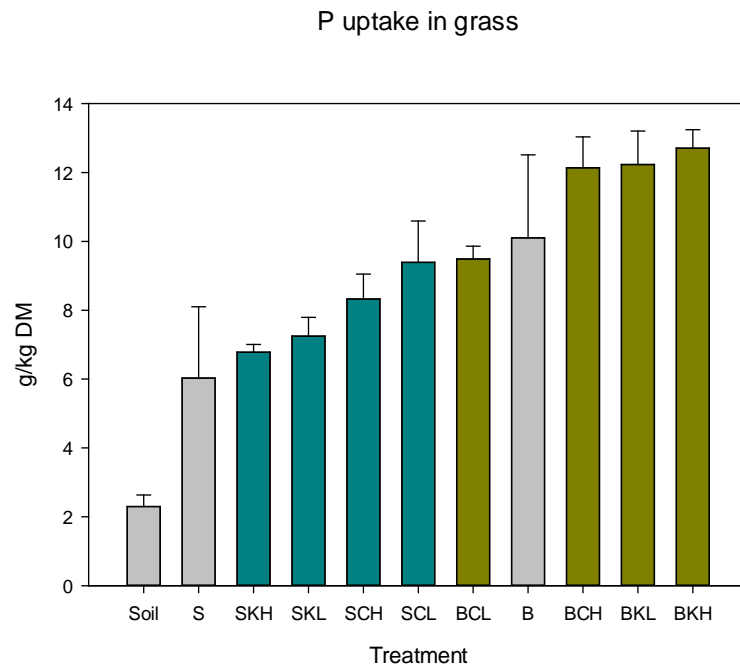
**Figure 12.** Total harvested dry matter. B = biochar (brown), S = sludge (blue), C = low pH treatment (compost leachate), K = high pH treatment (concrete), H = high, L = low dosage).



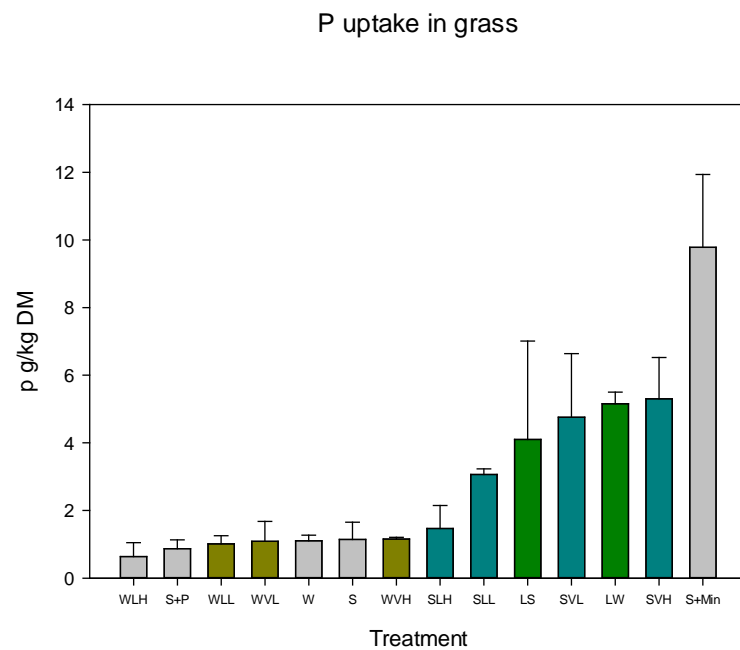
**Figure 13.** Grass harvest. Treatments: Grey = controls; S = sand + peat (no fertilizer), S + F: sand/peat + mineral fertilizer, S = fertilized with sludge, SVL: sludge + acid low conc., SVH = sludge acid high, W = wetland filter, W treatments as for sludge, LW = lupins + wetland, LS = lupin + sludge, L = lupin + mineral fertilizer (recycled).

sludge pots, there was a tendency to higher values in the low pH treatments (SC).

The P uptake was also higher in the pots fertilized with composted sludge (S), and in the low pH treatment (Figure 15). All pots with the lupin treatment (L)

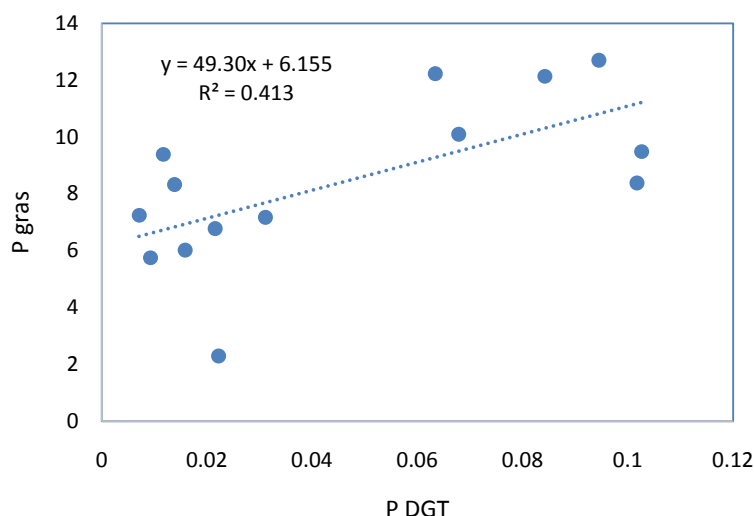


**Figure 14.** P uptake in grass from sludge and biochar. B = biochar (brown), S = sludge (blue), C = low pH treatment (compost leachate), K = high pH treatment (concrete), H = high, L = low dosage, grey = control.



**Figure 15.** P uptake in grass from sludge and wetland filters. Treatments: Controls = grey; S + P = sand + peat (no fertilizer), S + Min = sand/peat + mineral fertilizer, S = composted sludge (brown), SVL: sludge + acid low conc., SVH = sludge acid high, W = wetland filter (blue), W treatments as for sludge, L = lupins (green), LW = lupins + wetland, LS = lupin + sludge, L = lupin (recycled).

show relatively high P uptake. The P uptake in the Australian experiment was lower than the uptake in the Norwegian experiment (6 g to 12 g). The uptake of P



**Figure 16.** Correlation of the P concentrations in the DGTs and the grass.

both in grass and in the DGTs is clearly and significantly higher in the post-treated by lupins.

**Figure 16** shows a significant correlation between the P uptake measured in the DGTs and in the grass, with a correlation coefficient of  $R^2 = 0.41$ . P uptake as measured by the DGT is further correlated with K, Si while Fe, and uncorrelated with Ni, Cu and Ca.

#### 4. Conclusions

- The mean irrigation in the experiment was between 3 to 7 mm/day, the evapotranspiration between 2 and 3 mm/day in pots without and with vegetation;
- Grass P uptake was significantly higher in pots fertilized by biochar compared to pots fertilized by sewage sludge;
- Biochar responded better after pH treatment than sewage sludge;
- P uptake from composted sewage sludge was higher than from wastewater filter material;
- The sludge responded better with acidic treatment;
- The wetland filter did not respond to pH treatment;
- The lupin treatment showed a high uptake of P almost comparable to mineral fertilizer;
- The DGT samples produced approximately the same results as the direct measurements of the grass, although the correlation was not perfect;
- If phosphorus uptake is the priority, organic waste benefits from being turned into biochar before used as fertilizer.

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