Phosphorus availability of sewage sludge-based fertilizers determined by the diffusive gradients in thin films (DGT) technique

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Abstract

The plant-availability of phosphorus (P) in fertilizers and soil can strongly influence the yield of agricultural crops. However, there are no methods to efficiently and satisfactorily analyze the plant-availability of P in sewage sludge-based P fertilizers except by undertaking time-consuming and complex pot or field experiments. We employed the diffusive gradients in thin films (DGT) technique to quantify the plant P availability of various types of P fertilizers with a novel focus on sewage sludge-based P fertilizers. Mixtures of fertilizer and soil were incubated for 3 weeks at 60% water holding capacity. DGT devices were deployed at the beginning of the incubation and again after 1, 2, and 3 weeks. Two weeks of incubation were sufficient for the formation of plant-available P in the fertilizer/soil mixtures. In a pot experiment, the DGT technique predicted maize (*Zea mays* L.) biomass yield and P uptake significantly more accurately than standard chemical extraction tests for P fertilizers (e.g. water, citric acid, and neutral ammonium citrate). Therefore, the DGT technique can be recommended as a reliable and robust method to screen the performance of different types of sewage sludge-based P fertilizers for maize cultivation minimizing the need for time-consuming and costly pot or field experiments.

1 Introduction

The chemical form of phosphorus (P) applied to agricultural crops is a major determinant of its plant availability. Therefore, in order to improve the uptake of P by plants, substantial knowledge of the chemical form of P in fertilizers is necessary. This is particularly important for novel P fertilizers such as those obtained from waste materials (e.g. sewage sludge). Currently, several different processes for P recovery from waste water or sewage sludge are in development or already in operation (*Donatello* and *Cheeseman*, 2013; *Kabbe* et al., 2015). Previous pot experiments with sewage sludge-based P fertilizers demonstrated strong

variation in plant P availability (*Römer,* 2006; *Nanzer* et al., 2014; *Severin* et al., 2014; *Vogel* et al., 2015).

While pot experiments are useful for determining the plant availability of a P fertilizer, they are time-consuming and costly. Therefore, various simple chemical extraction methods are often used instead (*EU*, 2003; *Kratz* and *Schnug*, 2009; *Kratz* et al., 2016). These chemical extraction methods mainly differ in their pH values, and the type of organic acid used as the extractant (*EU*, 2003). However, despite the demand, there is currently no satisfactory extraction method available for determining the plant availability of sewage sludge-based P fertilizers. In pot experiments carried out with thermochemically treated sewage sludge ash, *Kratz* et al. (2010) found that neutral and alkaline ammonium citrate extraction methods were the most accurate predictors of plant P uptake and yield. However, for sewage sludge-based biochars, the neutral ammonium citrate extraction method did not show an adequate correlation to the results of pot experiments (*Steckenmesser* et al., 2017). This indicates that simple chemical extraction methods can be strongly influenced by the matrix of the fertilizer (*Axelrod* and *Greidinger*, 1979; *Braithwaite* et al., 1989).

For soil-P analysis, different chemical extraction methods (e.g. water, Olsen, CAL (= calcium acetate lactate), Colwell, Bray, Mehlich etc.) are often used (*Steffens*, 1994; *Mundus* et al., 2013; *Wuenscher* et al., 2015). More recently, several research groups (e.g. *Zhang* et al., 1998; *Menzies* et al., 2005; *Mason* et al., 2010; *Tandy* et al., 2011; *Moody* et al., 2013; *Speirs* et al., 2013; *Mundus* et al., 2013; *Six* et al., 2014; *Santner* et al., 2015; *Davison*, 2016) have shown that the diffusive gradients in thin films (DGT) method has a much better correlation to plant-available P in soils than standard chemical extraction tests. The DGT device consists of a binding layer, a diffusion gel and a filter (to protect the gel) in a plastic piston (*Davison* et al., 2016). The dissolved P fraction of the soil solution from moist soil samples diffuses through

the filter and diffusion gel (normally for 24 h), and is subsequently adsorbed to the binding layer. The amount of P accumulated on the binding layer depends on the diffusion of P through the diffusion layer and the resupply of labile P from the solid soil phase. This amount of adsorbed P on the binding layer is then used to calculate the diffusion-limited, time-averaged concentration of P, which we used as indicator for the plant availability of the soil-P.

The aim of our work was to develop a reliable and robust method to quantify the plant-available P in sewage sludge-based P fertilizers by adopting the DGT technique and deploying the DGT device on various fertilizer/soil-mixtures. Furthermore, we compared the DGT method with different commonly applied chemical extraction methods to quantify the plant-available P in soil fertilized with various sewage sludge-based P fertilizers. The plant-available P quantified with these methods was then compared to the yield and P uptake of maize grown on the same amended soils.

2 Material and methods

2.1 P-fertilizer production

Different sewage sludge-based P fertilizers were prepared by low-temperature conversion and thermochemical post-treatment of sewage sludge sourced from two wastewater treatment plants in Germany. One sewage sludge was simultaneously precipitated with $FeCl_2$ and directly fermented, which resulted in an anaerobically stabilized sludge. The other sewage sludge originated from a waste water treatment plant with enhanced biological phosphorus removal (EBPR). This sludge was aerobically stabilized followed by P-precipitation with $Al_2(SO_4)_3$. Both sewage sludges were converted to biochars by low-temperature conversion in a lab-scale reactor at 400-500°C (*Weber* et al., 2014; *Steckenmesser* et al., 2017). Hereafter, the sludges will be referred to as B_{chem} and B_{bio}, respectively. The resulting biochars were used either directly for the pot experiment or further thermochemically processed at 950°C for

approximately 30 min in a rotary furnace (Thermal Technology, RT1700, corundum tube, Bayreuth, Germany) to improve the fertilizer performance of the material as follows:

1. Treatment of B_{chem} with magnesium chloride ($MgCl_2$; Sigma-Aldrich, Steinheim, Germany) under oxidizing (air) conditions (*Adam* et al., 2009; *Vogel* and *Adam*, 2011; hereafter the product is referred to B_{chem}-Mg).

2. Treatment of B_{bio} with sodium sulfate (Na_2SO_4; Applichem, Darmstadt, Germany) under reducing conditions (*Stemann* et al., 2015; *Herzel* et al., 2016; *Vogel* et al., 2016; hereafter the product is referred to B_{bio}-Na).

3. Treatment of B_{chem} first a gaseous chloride donor (hydrochloric acid) to remove heavy metals (*Vogel* and *Adam*, 2011), followed by addition of Na_2SO_4 (under reductive conditions) to enhance the P plant-availability (hereafter the product is referred to B_{chem}-Cl+Na).

The P-bearing mineral phases of the sewage sludge-based P fertilizers were determined using X-ray diffraction (*Steckenmesser* et al., 2017). The biochar B_{chem} mainly contained inorganic pyro- and polyphosphates. After thermochemical treatment with $MgCl_2$ (B_{chem}-Mg), chlorapatite ($Ca_5(PO_4)_3Cl$) and stanfieldite ($Ca_4Mg_5(PO_4)_6$) were detected as P phases. The HCl/Na_2SO_4 treatment (B_{chem}-Cl+Na) led to the formation of calcium sodium phosphate ($CaMgNa_2(PO_4)_2$). For the biochar B_{bio}, meta- or short polyphosphates were assumed. After treatment with Na_2SO_4 (B_{bio}-Na), calcium sodium phosphate ($Ca_{13}Mg_5Na_{18}(PO_4)_{18}$) and whitlockite were detected.

2.2 Pot experiment

Three kg of a subsoil from a brown earth derived from loess were mixed with 3 kg of quartz sand, to decrease the P mass fraction (P_{Total}= 180 mg kg^{-1}, P_{CAL}= 10 mg kg^{-1}, pH in 0.01 M $CaCl_2$ = 6.7 of mixture), was used for the pot experiment. The experiment consisted of six

treatments; five sewage sludge-based P fertilizers and one triple superphosphate (TSP) treatment. A control with no added P (P0) was also included. Four replicates were used per treatment (also for P0), and the test plant was maize (*Zea mays* L. cv. Amadeo). Based on pre-tests, a P application rate of 0.6 g per pot was adequate to achieve maximum shoot biomass of maize plants after 30 d of cultivation. Therefore, the soil/sand mixture was fertilized with 100 mg P kg^{-1} soil of each P fertilizer as well as 250 mg N (as NH_4NO_3), 300 mg K (50% KCl + 50% K_2SO_4), and 150 mg Mg ($MgSO_4$) per kg soil. Micronutrients were added as an aqueous solution containing 20 mg Mn ($MnSO_4$), 10 mg Zn ($ZnSO_4$), 5 mg Cu ($CuSO_4$), 0.5 mg B (H_3BO_3), and 0.11 mg Mo ($(NH_4)_6Mo_7O_{24}$) per kg of soil.

The pot experiment was conducted in Mitscherlich pots (height: 18 cm, diameter: 20 cm) in a growth chamber with 16 h light at 25°C, 8 h dark at 18°C, and light intensity of 150 W m^{-2}. After 1 week of pre-germination, three seeds were sown in each pot and harvested after 30 d. Pots were watered daily to maintain 60% water-holding capacity (WHC). The above-ground biomass was harvested from each pot, weighed (fresh weight) and then chaffed. Biomass yield was determined after drying the plant material at 105°C. The P concentration of the dried plant material was determined using ICP-OES (Varian 720-ES, Waldbronn, Germany; wavelength 213.6 nm) after digestion with nitric acid, perchloric acid, and sulfuric acid under an increasing temperature (up to 220°C) for 6 h. The derived residue was dissolved in 5 M HCl and filtered. Homogenized soil samples were collected from each pot after harvest and air-dried at room temperature for 1 month before analysis.

2.3 Chemical extraction tests for soils and fertilizers

Homogenized subsamples of each treatment of the post-harvest soils were extracted in triplicate using three chemical extractants to quantify the extractable P: water (soil : solution ratio 1 : 10, 60 min), calcium acetate lactate (CAL, *Schüller*, 1969; soil : solution ratio 1 : 20,

120 min), and sodium bicarbonate (*Olsen* et al., 1954; soil : solution ratio 1 : 20, 30 min). The P fertilizers were also extracted with water ($P_{W;}$ fertilizer : solution ratio 1 : 100, 30 min), 2% citric acid ($P_{CIT;}$ fertilizer : solution ratio 1 : 100, 30 min) and neutral ammonium citrate ($P_{NAC;}$ fertilizer : solution ratio 1 : 166, 60 min) and carried out according to the EU regulation No. 2003/2003 (*EU*, 2003) in triplicate. The extracted P concentrations from these chemical extraction tests were analyzed using ICP-OES (Thermo iCAP 7000, Dreieich, Germany).

2.4 DGT experiments

Post-harvest soil samples were also analyzed with DGT devices equipped with a ferrihydrite binding layer. After a 24 h conditioning period of the soil at 60% of the WHC, the soils were brought to 100% WHC, transferred into the DGT devices and deployed for 24 h at 25°C (*Davison*, 2016). Following the extraction of P from the binding layer with 1 M HNO_3, P concentrations were analyzed with ICP-MS (Thermo iCAP Q, Dreieich, Germany) and used to calculate the DGT P (P_{DGT}). The DGT measurements were carried out in duplicate.

Additional DGT measurements were also performed with the fertilizer/soil mixtures in order to predict the performance of the various P fertilizers using a simple and rapid test. The different P fertilizers were mixed with the same soil/sand mixture and nutrients (N, K, Mg, S, micronutrients) that were used for the pot experiment (see above). Specifically, 15 g of the soil/sand mixture were mixed with 1.5 mg P from the respective P fertilizer for each experiment (= 100 mg P per kg of soil) in 50 mL plastic containers. These DGT measurements were also carried out in duplicate. Other nutrients were added to the fertilizer/soil-mixtures using the same nutrient : soil ratio as applied in the pot experiment. The soils were maintained at 60% WHC using deionized water and stored at 22°C. At the start of the incubation experiment, and after 1, 2, and 3 weeks, DGT devices were deployed. As with the pot experiment, the water content was increased to 100% WHC before DGT deployment.

2.5 Statistics

For statistical analysis of the pot experiment, chemical extraction tests and DGT experiments, an ANOVA and a Tukey test were performed ($P < 5\%$) with the software R (version 3.4.0).

3 Results

3.1 Plant P availability in soils from pot experiment

B_{chem}-Cl+Na (49% P uptake relative to TSP) and B_{bio} (44%) showed the highest dry matter yield and P uptake by maize of the sewage sludge-based P fertilizers in the pot experiment (Tab. 1), followed by B_{bio}-Na (31%) and B_{chem} (26%). On the other hand, B_{chem}-Mg (7%) only resulted in comparable yield and P uptake to the P0 treatment (i.e. no P added; 4%). With the extraction tests, B_{bio}-amended soils after harvest showed the highest extractable P values of the sewage sludge-based P fertilizers with all three tests (water, Olsen, and CAL extractions), with CAL showing an even higher value than TSP. Additionally, the P values of B_{chem}-Cl+Na were relatively high for these three chemical extraction tests. Soils with B_{chem}-Mg, B_{bio}-Na and P0 treatments had the lowest values of extractable P. Figure 1 shows the plot of P uptake by maize plants compared with P values obtained with water, Olsen and CAL extractions in the post-harvest soils of the treatments with the applied P fertilizers. Phosphorus uptake by maize showed a modest correlation with the chemical extraction tests in the pot experiment, with the Olsen extraction performing the best out of the three extraction methods. Furthermore, the standard deviations of the chemically extracted P concentrations were high for some soil samples (%RSD values range from 1 to 52 %). In contrast, the DGT results showed a superior correlation with P uptake by maize with much lower deviation of the measured values (RSD < 10%) compared to the results of the chemical extraction tests.

3.2 Chemical extraction tests of applied P-fertilizers

The water-extractable P (P_W) was very low for the sewage sludge-based P fertilizers, with the highest P_W observed for the sodium-treated products: B_{bio}-Na (5%) and B_{chem}-Cl+Na (2%; Tab. 2). The P extracted with citric acid (P_{CIT}) and neutral ammonium citrate (P_{NAC}) was much higher for all applied P fertilizers, with the highest P_{CIT} and P_{NAC} observed from B_{chem} and B_{bio}-Na, respectively. Figure 2 shows that there is a modest correlation between the P_W of the applied P fertilizers and the P uptake by maize in the pot experiment with the same P fertilizers, even if the values for the sewage sludge-based P fertilizers were relatively small. In contrast, the extractable P of P_{CIT} and P_{NAC} showed no correlation with the P uptake by maize.

3.3 DGT technique applied to fertilizer/soil mixtures

Without incubation the application of the two B_{bio} products showed the highest DGT P (P_{DGT}) values for the fertilizer/soil mixtures of the sewage sludge-based P fertilizers (Tab. 2). The P_{DGT} values increased after 1 and 2 weeks of incubation (except with B_{chem}-Mg which did not change). The P_{DGT} values of B_{bio}-Na reached their maximum after 1 week, while the other sewage sludge-based P fertilizers required 2 weeks of incubation to reach their highest P_{DGT}. In contrast, the P_{DGT} values of TSP decreased after the first week of incubation and remained at this level. In addition, Fig. 3 shows the P uptake by maize in relation to the results from the incubated fertilizer/soil DGT experiments in the corresponding pot experiment. After 2 weeks of incubation, the correlation is better than after 1 week of incubation. The P_{DGT} after 3 weeks of incubation and the correlation with P uptake by maize were not significantly different to those after 2 weeks.

4 Discussion

4.1 Comparison of plant P availability of soils from pot experiment

The results of the pot experiment show that different treatments of sewage sludge resulted in P fertilizers with different plant P availability. During the pot experiment, the plants did not take up all of the applied P. The remaining P in the amended soils can be used as an indicator for the P-fertilizer performance. For all chemical extraction tests, the soil amended with the fertilizer B_{bio} showed the highest extractable P of the sewage sludge-based P fertilizers. It is assumed, that the biochar B_{bio} contains—in contrast to B_{chem}—more soluble meta- and short polyphosphates because it originated from a waste-water treatment plant with enhanced biological P removal (*Stratful* et al., 1999; *Steckenmesser* et al., 2017). *Torres-Dorante* et al. (2006) showed that meta- and short polyphosphates have a high plant P availability. In contrast, B_{bio}-Na, the product of thermochemical treatment of B_{bio} Na_2SO_4, allowed a much lower biomass yield and P uptake than B_{bio}. This may be attributed to the decomposition of meta- and polyphosphates during the treatment process. In contrast to the chemical extraction methods, the DGT method showed an excellent correlation with the P uptake by maize. This correlation is in agreement with other studies (*Menzies* et al., 2005; *Six* et al., 2014; *Wang* and *Chu*, 2015; *Christel* et al., 2014, 2016a) that found a good correlation between the amount of added fertilizer P and adsorbed P in soil determined by DGT methods. This finding confirms the validity of using the DGT approach for P-fertilizers derived from sewage sludge.

Furthermore, the standard deviation of the respective P values were much higher for the chemical extraction tests than for P_{DGT}. For the DGT experiments, a greater mass of soil was used compared to that for the chemical extraction tests (15 g instead of 5 g) which may be an explanation for the lower standard deviation P_{DGT}. In addition, different P fertilizers can change the soil pH in different ways, which can negatively affect several soil P extraction methods (*Wuenscher* et al., 2015). In contrast, the DGT method is unaffected by pH within the range 3-

9 (*Mason* et al., 2005), because it uses water instead of buffer solutions as extractant. Therefore, the plant-available P can be analyzed at the native soil pH, as confirmed during P adsorption tests with iron oxide strips, which act as the DGT binding layer, in soils (*Uusitalo and Yli-Halla*, 1999; *Mundus* et al., 2013).

4.2 Chemical extraction tests of sewage sludge-based P fertilizers

The extractable P in water (P_W) of the sewage sludge-based P fertilizers is very low (≤ 5%) in contrast to TSP (96%). However, the P_W shows the best correlation of the chemical extraction tests with the P uptake by maize in the pot experiment ($R^2 = 0.73$). Hence, this good correlation is strongly influenced by the P_W value of TSP. In contrast, the extractable P is much higher in citric acid (P_{CIT}) and neutral ammonium citrate (P_{NAC}), but there was no correlation with the P uptake by maize in the pot experiment. This demonstrates that these chemical extraction tests are not predictive for P plant uptake for various types of sewage sludge-based P fertilizers. *Steckenmesser* et al. (2017) showed a strong correlation for the P uptake and biomass yield by maize treated with chemically precipitated sewage sludge-based P fertilizers with the extractable P in 2% formic acid. In contrast to this, *Kratz* et al. (2010) recommend the P_{NAC}-extractable P as the best indicator for plant P availability based on modified Neubauer trials with summer rye grown in soil amended with sewage sludge ash based P-fertilizers. Thus, there is no universally applicable chemical extraction test which can quantify the plant P availability of various sewage sludge-based P fertilizers.

4.3 Estimation of plant-available P in P fertilizers with the DGT technique

The DGT results from the incubated fertilizer/soil mixtures have, in contrast to the chemical extraction tests, a much better correlation with the P uptake by maize in the pot experiment. For all applied P fertilizers, 2 weeks of incubation seemed to be sufficient for the formation of plant-available P in the fertilizer/soil mixtures. In contrast to the sewage sludge-

based P fertilizers the P_{DGT} value of TSP/soil incubation decreased in the first week. This is easily rationalized given that P from TSP ($Ca(H_2PO_4)_2 \times H_2O$) is highly soluble, and is slowly converted to water-insoluble $CaHPO_4 \times 2\ H_2O$ over the incubation period (*Vogel* et al., 2013). Therefore, it should be noted that direct application of DGT to analyze P fertilizers is not informative because water-soluble P fertilizer (e.g. TSP) can simply saturate the binding layer too quickly, while water-insoluble P fertilizers (e.g. sewage sludge (ash)-based materials) release only negligible amounts of P without prior incubation in soil (*Christel* et al. 2016b).

However, the correlation between P uptake and P_{DGT} values was not as strong as measured from the actual soils in the pot experiment, even though it was the same soil with the same micro and macro-nutrients. This could be due to the absence of plant roots that lead to a lower mobilization of P from the applied P fertilizers in the fertilizer/soil mixture. Furthermore, the R^2 values of the correlations in Fig. 3 are highly dependent on the P_{DGT} value of TSP. Thus, the DGT approach will not substitute pot or field experiments completely in all scenarios, but does offer a time and cost-effective screening method to support the measurement of P-fertilizer performance.

Finally, the ferrihydrite DGT binding layer can adsorb only up to approximately 50 µg of P and larger amounts of P in fertilizer/soil mixtures could possibly saturate the binding layer, even with slow-releasing, water-insoluble P fertilizers. Therefore, binding layers with higher capacities may be used, such as titanium dioxide (*Panther* et al., 2010), zirconium oxide (*Ding* et al., 2010) or magnesium carbonate (*Xie* et al., 2016). An alternative is a shorter deployment time of the DGT devices for incubated fertilizer/soil mixtures.

5 Conclusions

In this paper, we have demonstrated the potential of the DGT approach to become a key method for the determination of plant-available P in sewage sludge-based P fertilizers. The P value determined with DGT was strongly correlated with the P uptake by maize cultivated with a range of sewage sludge-based P fertilizers. Furthermore, by adopting the DGT approach an improved assessment of the plant-available P of various P fertilizers was made, compared to the conventional chemical extraction tests. We conclude that while the DGT method cannot completely substitute pot experiments, it is a suitable method for time and cost-effective screening of P-fertilizer performance.

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456

457 **Table 1:** Dry matter yield and P uptake by maize and water, Olsen and CAL extractable P and DGT P results from the soils after harvest in the
458 corresponding pot experiment. Different letters within each column indicate significant differences between treatments according ANOVA and
459 Tukey test ($p<0.05$).

	Dry matter yield (g)	**P uptake** (mg P/pot)	**Water extraction** (mg/kg)	**Olsen extraction** (mg/kg)	**CAL extraction** (mg/kg)	**DGT** (µg/l)
P0	2.5 ± 0.3 a	2.5 ± 0.4 a	5.4 ± 0.3 bc	8.5 ± 2.4 ab	7.6 ± 1.0 a	3.3 ± 1.1 a
B_{chem}	13.5 ± 1.4 b	18.1 ± 1.7 b	6.2 ± 0.9 bc	8.9 ± 4.6 ab	35.6 ± 2.8 b	27.8 ± 4.4 b
B_{bio}	20.6 ± 0.9 c	30.5 ± 2.3 c	7.2 ± 1.4 bc	15.7 ± 3.2 b	49.2 ± 9.4 b	54.7 ± 5.0 c
B_{chem} -Mg	2.8 ± 0.6 a	4.5 ± 1.0 a	0.1 ± 0.0 a	3.6 ± 0.0 a	35.5 ± 7.9 b	10.4 ± 2.7 a
B_{chem}-Cl+Na	20.6 ± 3.4 c	33.4 ± 7.2 c	6.7 ± 0.0 bc	9.1 ± 3.0 ab	42.1 ± 12.0 b	62.4 ± 3.9 c
B_{bio}-Na	13.8 ± 1.2 b	21.6 ± 3.5 b	2.1 ± 0.2 ab	7.6 ± 0.1 ab	25.7 ± 2.7 ab	46.4 ± 3.2 b
TSP	29.2 ± 0.8 d	68.8 ± 5.8 d	8.5 ± 2.4 c	22.8 ± 5.1 c	38.1 ± 10.6 b	135.6 ± 0.6 d

460

461 **Table 2:** P_W, P_{CIT} and P_{NAC} extracted from the applied P-fertilizers and P_{DGT} results from incubated fertilizer-soil mixtures (direct, one, two and
462 three weeks). Different letters within each column indicate significant differences between treatments according ANOVA and Tukey test ($p<0.05$).

	P_W (%)	**P_{CIT}** (%)	**P_{NAC}** (%)	**P_{DGT} direct** (µg/l)	**P_{DGT} 1 week** (µg/l)	**P_{DGT} 2 weeks** (µg/l)	**P_{DGT} 3 weeks** (µg/l)
B_{chem}	0 ± 0 a	93 ± 6 b	30 ± 1 a	17.8 ± 1.4 a	31.1 ± 2.1 ab	61.2 ± 2.7 b	56.1 ± 8.5 b
B_{bio}	1 ± 0 a	33 ± 3 a	25 ± 5 a	49.0 ± 10.6 a	133.2 ± 7.1 c	181.0 ± 6.9 c	177.5 ± 36.1 c
B_{chem}-Mg	0 ± 0 a	73 ± 2 bc	53 ± 2 b	9.9 ± 6.9 a	7.4 ± 0.4 ab	7.8 ± 0.5 a	11.5 ± 0.0 a
B_{chem}-Cl+Na	2 ± 0 a	58 ± 14 ab	31 ± 5 a	33.1 ± 5.6 a	67.2 ± 9.2 abc	90.6 ± 7.6 b	80.7 ± 6.4 b
B_{bio}-Na	5 ± 0 a	66 ± 0 b	70 ± 3 c	78.2 ± 22.8 a	112.7 ± 11.7 bc	106.3 ± 10.8 b	112.2 ± 14.8 bc
TSP	96 ± 5 b	100 ± 0 c	49 ± 0 b	1193.1 ± 145.7 b	678.2 ± 36.8 d	542.9 ± 27.1 d	664.4 ± 18.4 d

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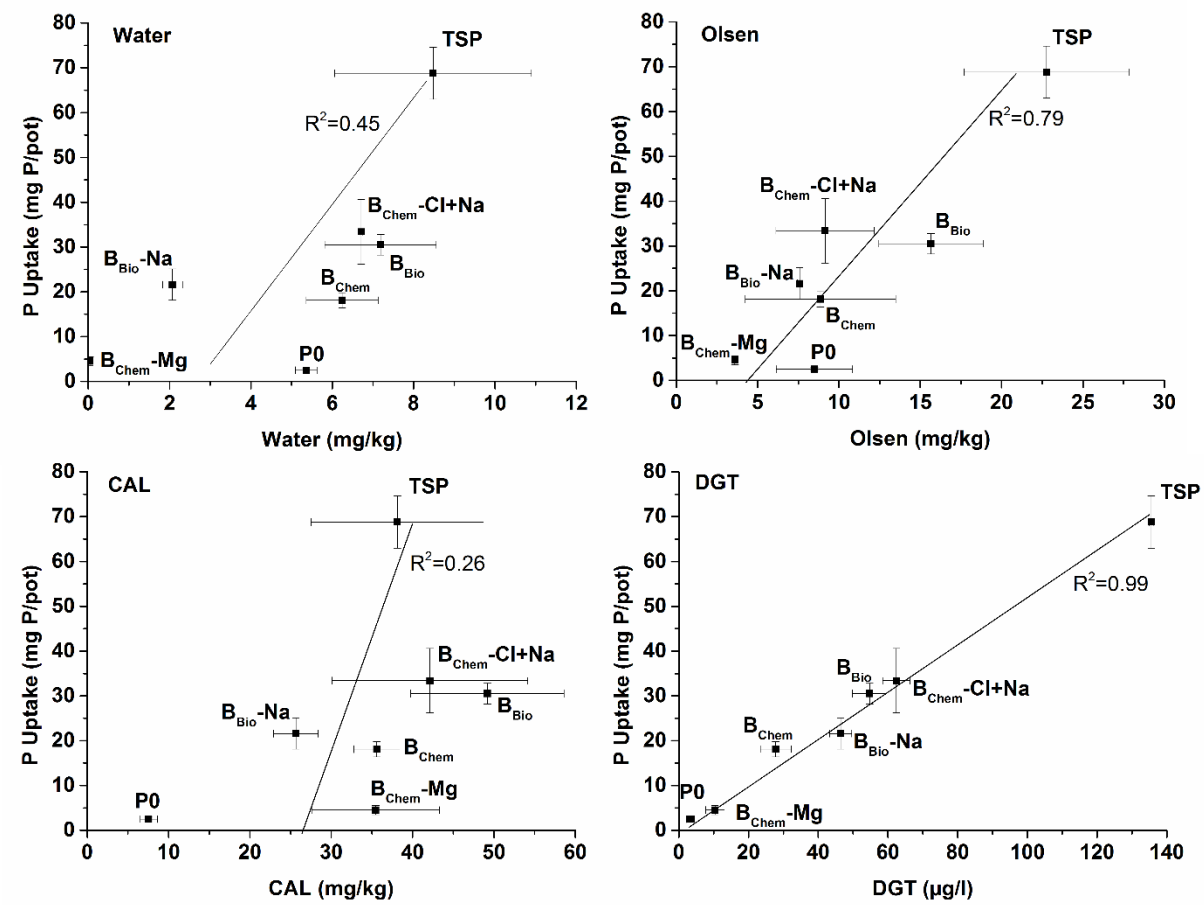

Figure 1: Water, Olsen and CAL extractable P and DGT P results of the fertilized soils with different sewage sludge based P-fertilizers, analyzed after harvest, in relation to the P uptake by maize in the corresponding pot experiment. The vertical and horizontal lines show the standard deviation of each point.

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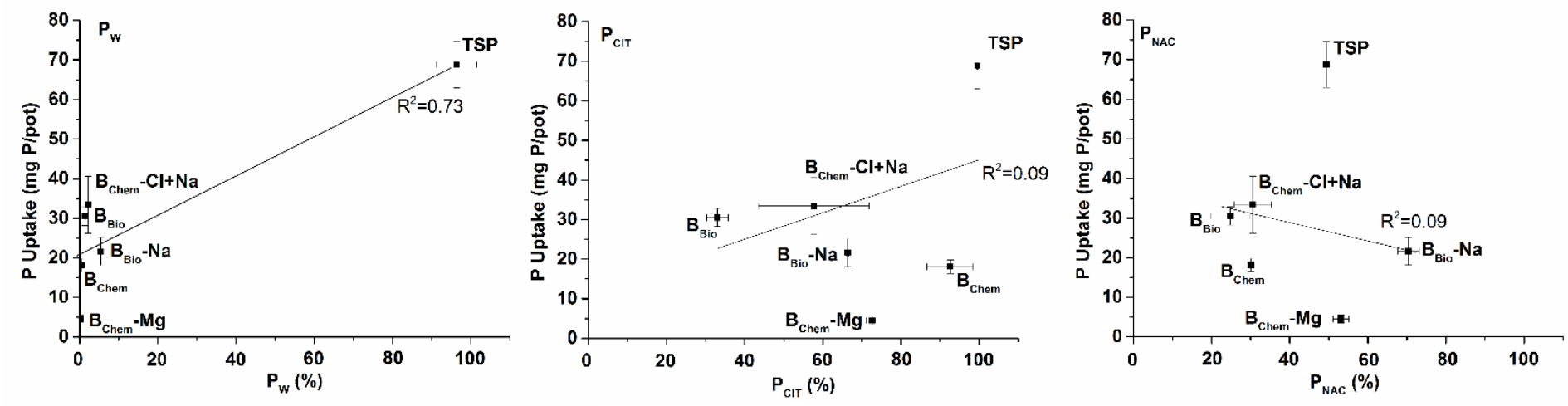
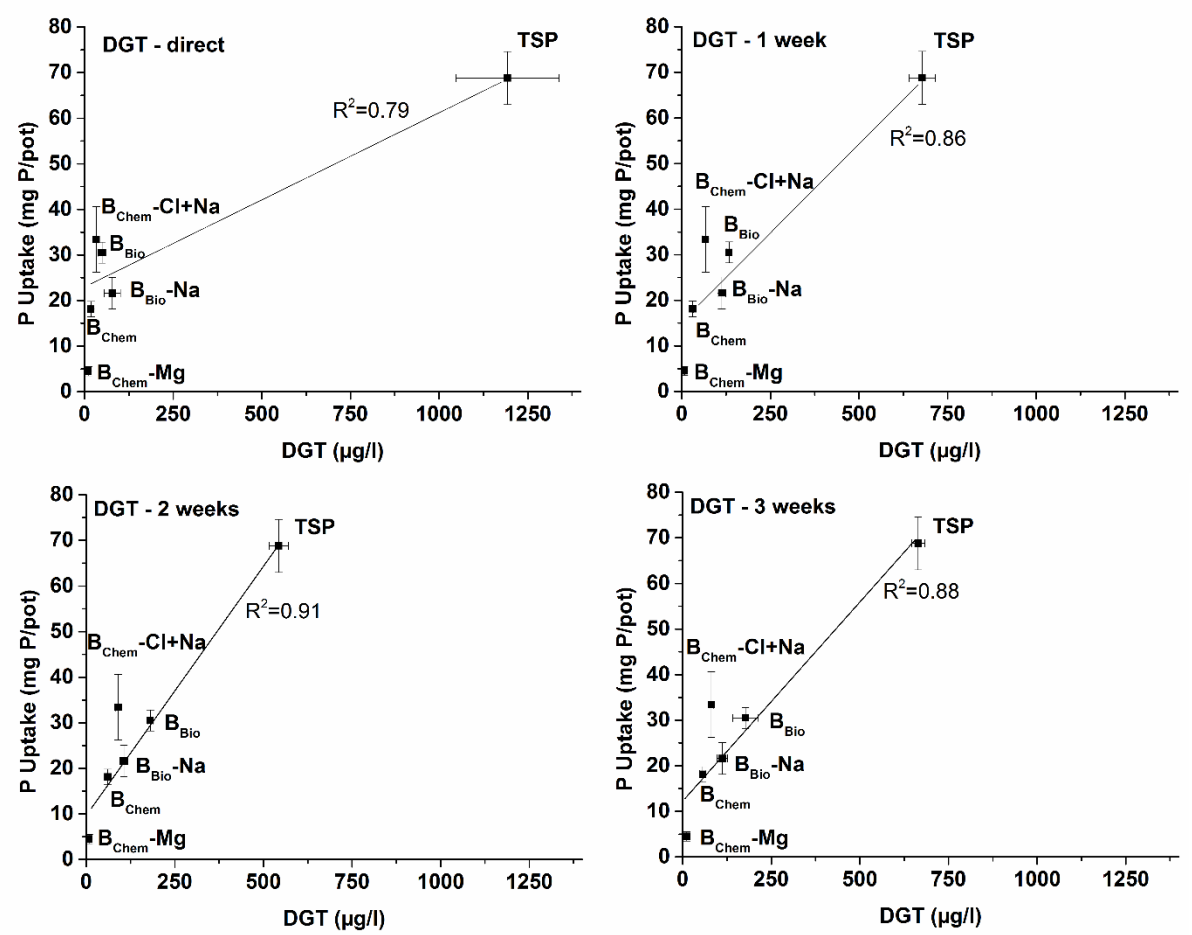


473

474 **Figure 2:** Water (P_W), citric acid (P_{CIT}) and neutral ammonium citrate (P_{NAC}) extractable P of the applied P-fertilizers in relation to the P uptake by

475 maize in the corresponding pot experiment with these fertilizers. The vertical and horizontal lines show the standard deviation of each point.

476



477

478 **Figure 3:** P_{DGT} results of the incubated fertilizer-soil mixtures (direct, one, two and three

479 weeks) in relation to the P uptake by maize in the corresponding pot experiment with these

480 fertilizers. The vertical and horizontal lines show the standard deviation of each point.

481